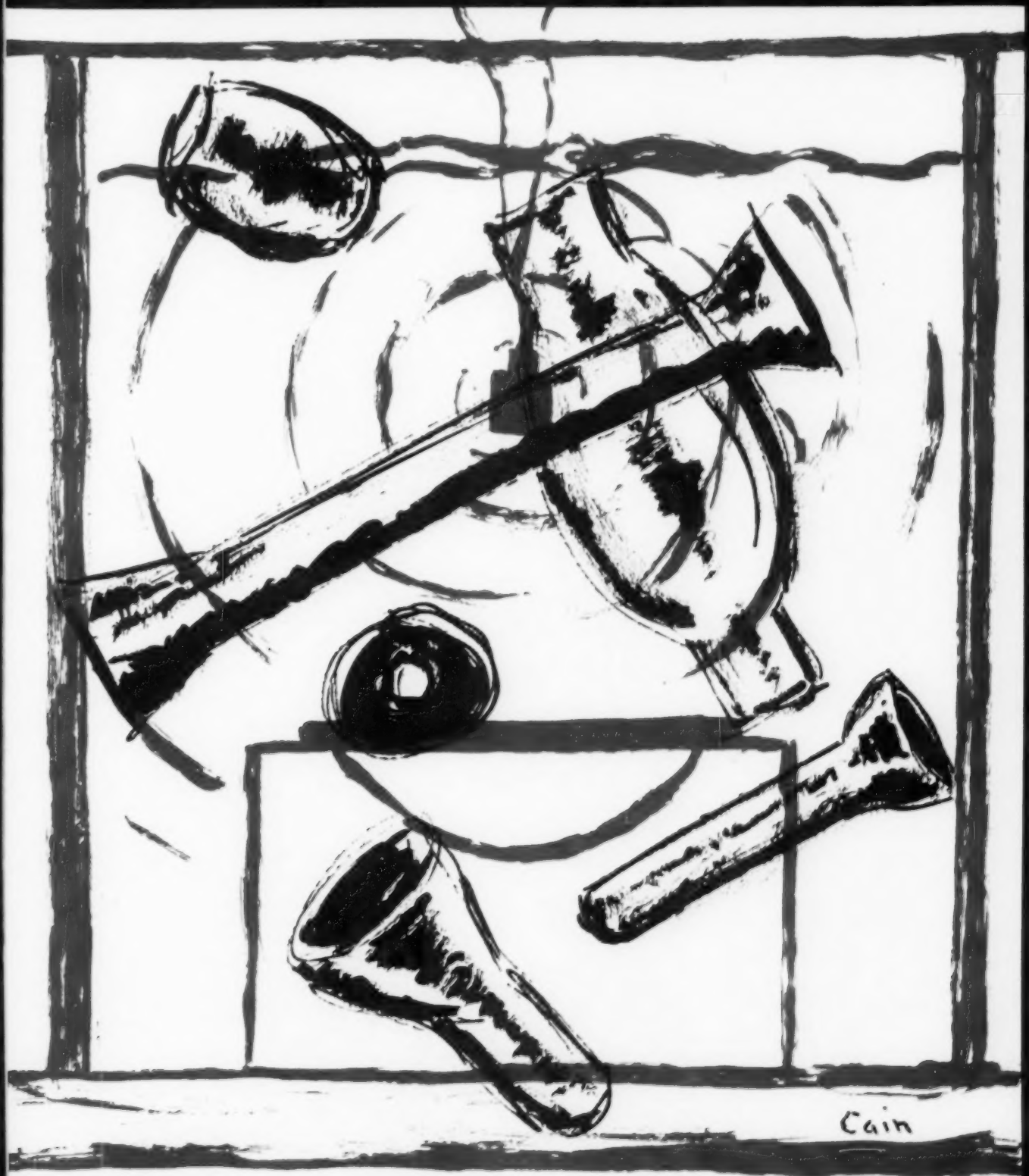
JOURNAL OF

METALS

SEPTEMBER 1960



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METALS

Vol. 12, No. 9
September, 1960

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EDITORIAL STAFF: Editor in Chief, F. Weston Starratt; **Associate Editor,** Harvey Braun, **Production Editor,** Otto Johnson; **News Editor,** William Leggitt; **Editorial Assistant,** C. M. Cardon.
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personnel

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- When making application for a position, include eight cents in stamps for forwarding application to the employer and for returning, when possible.

—MEN AVAILABLE—

All those listed under "Men Available" have membership in the AIME.

Metallurgist, PhD. Broad background in physical metallurgy, ferrous and non-ferrous research and production experience. Foreign born, European and American educated. Administrative interests. Several foreign languages. Will relocate, prefer New York area. M-239.

Metallurgical Engineer, M.S., Met. Eng., age 36. Experienced director engineering and manufacturing. Experience in specialized foundry, powder metallurgy, primary working of metals. Desire responsible position with progressive company leading to management position. Location: open. M-1150-Chicago.

Metallurgical Engineer, B.S. Met. Eng., age 30. Four years research and development department of large integrated steel mill. Responsibilities include conduction and direction of both laboratory and production type experimentation. Location: Midwest. M-1151-Chicago.

Metallurgist, B.S., Met. Eng., age 41. Ten years metallography of valve and jet alloys in automobile and aircraft. Consultant and designer for mitigation of soil and water corrosion, heat treatment of carbon steel. Location, Midwest or West. M-1152-Chicago.

Senior Metallurgical Engineer, B.S. ChE. Experienced refractory metals, high temperature testing-arc melting-casting alloys. Prefer New York Metropolitan area. M-240.

Chief or Supervising Metallurgist, physical or process, B.S. Physics, age 35. Nine years R&D, production soft magnetic materials; four years supervisor, customer service, technical administration. Seeking line management position embracing R&D, production, and sales utilizing specialized experience. Location, open. M-241.

Assistant Research, Development, Sales. MetE, age 38. Recent student, four years experience maintenance of aircraft, desires position as engineering aide, technical assistant or sales in chemical or metallurgical engineering or personnel, industrial relations. Salary open. Prefer San Francisco Bay Area. Home: San Francisco Peninsula. Se-1064.

Metallurgist. MetE, 27. Four years research and development in new smelting practices, modernization of operation, trouble shooting, new uses for lead and lead alloys for mining company. Military completed. Se-1380.

—POSITIONS OPEN—

Welding Engineer, graduate mechanical or metallurgical engineer, with five to eight years experience in welding engineering, and with at least two years in charge of a welding engineering group performing welding engineering, research and development. Must have good working knowledge of major welding process and a good foundation in ferrous metallurgy, in particular the welding and application of metal for high temperature, high pressure service. Should have technical writing skill and ability to speak effectively before moderately large groups. Must be capable of effectively supervising engineers and technicians performing both development work and shop welding control. Salary, $10,000-$12,000 year. Company pays placement fee. Location: Pennsylvania. W9403.

Engineers experienced in the iron and steel industry. (a) Furnace and Heat Engineer, graduate mechanical or metallurgical, with an intensive background in operation of steel mills, particularly in reference to furnace design, heating engineering, furnace operations, etc. Salary, $13,000-$15,000 year. (b) Rolling Mill Engineer, graduate metallurgical, or mechanical engineer, with strong background in steel mill operation. Experience must include extensive exposure to rolling mill design and equipment installation. Salary, $13,000-$15,000 year. Company pays placement fees. Location: New York, N. Y. W9399.

Metallurgist, young, need not be graduate, for company in precision casting, foundry and heat treating operations. Primarily to solve problems involving all alloys. Salary, $6000-$7800. Company will negotiate fee. Location: New York, N. Y. W9397.

Metallurgist, Physical Metallurgical Research, graduate, with M.S. preferred, with two to four years experience in physical metallurgical research, but will consider outstanding recent graduates. Excellent background would include physical metallurgical research and development experience with high strength steel alloys. Experience developing magnesium alloys also helpful. Experience in the aircraft-missile field helpful. Salary to $8000 year. Location: northern New Jersey. W9381.

Project Engineer (Trainee), B.S. in mechanical or metallurgical engineering, with none to two years experience, with basic knowledge of engineering principles and ability to apply these principles to routine problems. One year training program designed to provide familiarization and training in basic engineering principles of each of the division product lines. Will be assigned to one of four engineering groups, with advancement toward project or product engineer. Salary to $6300 year. Training locations: northern New Jersey, Missouri, Chicago, Ill., Ohio or Alabama. W9380.

Physical Metallurgist with two years plant experience for processing and laboratory control in aluminum casting and rolling operations. Location: Pennsylvania. W9343.

Chief Metallurgist and Quality Control Supervisor, graduate metallurgist, with five to ten years experience, to take charge of R&D metallurgical laboratory, foundry and product inspection. Should have knowledge of melting practices, sand control, centrifugal casting and particularly copper base alloys. Salary, $10,000-$15,000 year. Location: Ohio. W9328.

Section Head or Manager, Ceramic Research, advanced degree, PhD. preferred, in materials engineering, ceramics, metallurgy or physical chemistry; at least three years of research or development work in a ceramic field is required. Will plan and supervise studies on the properties of glasses, enamels, whitewares, and ceramic materials, reporting to Director of Research. Salary, $11,000-$14,500 year. Company may negotiate placement fee. Location: Pennsylvania. W9327.

Chief - Metallurgist - Mine Superintendent, graduate, over 35, with five to ten years experience, for a manganese, silver-lead-zinc mine. Small operation employing from 20 to 80 men. Location: West. W9314.

Project Engineer, graduate chemical, ceramic or metallurgical, for manufacturer of insulation and building products, with five years research experience in related industry, for product development work. Salary from $9000 year. Excellent opportunity. Location: central New Jersey. W9282.

Production Engineer, 25-35, graduate mechanical, with a metallurgy minor desired, with practical experience in production engineering involving the metal making or metal fabricating industry; equipment design experience; familiarity with various kinds of metals and their characteristics; some knowledge of factors affecting the structure and constitution of alloys. Under direction, will be responsible for providing technical service to all departments in materials division for the purpose of improving and development, maintenance, and economical utilization of division's facilities to produce maximum quality at minimum cost. Location: Connecticut. W9268.

Pilot Plant Superintendent, MetE or MinE, 30-45. Experienced in pilot plant ore dressing operation (heavy media, jigging, spirals, flotation, magnetic separation) for iron ore operation. Working knowledge of Spanish desirable. Possible family housing after employment, after two or three months single status. For an open pit mine operation. About $9600 plus benefits. Location: Peru, S. A. Sj-5399-R.

Sales, sales background, to 38. Should have industrial sales experience of some product to some shops in the East Bay Area. Will sell bar and some strip stainless steel, particularly for tool specialties, to machine shops and factories. For a branch office of a well established steel manufacturer. Four to six weeks' training period required in Eastern U. S. Car required. Base salary $5400-$6000 to start plus quarterly and annual commissions. Potential for $8000-$10,000. Placement fee reimbursed after one year employment. San Francisco Bay Area men only. Sj-5313.

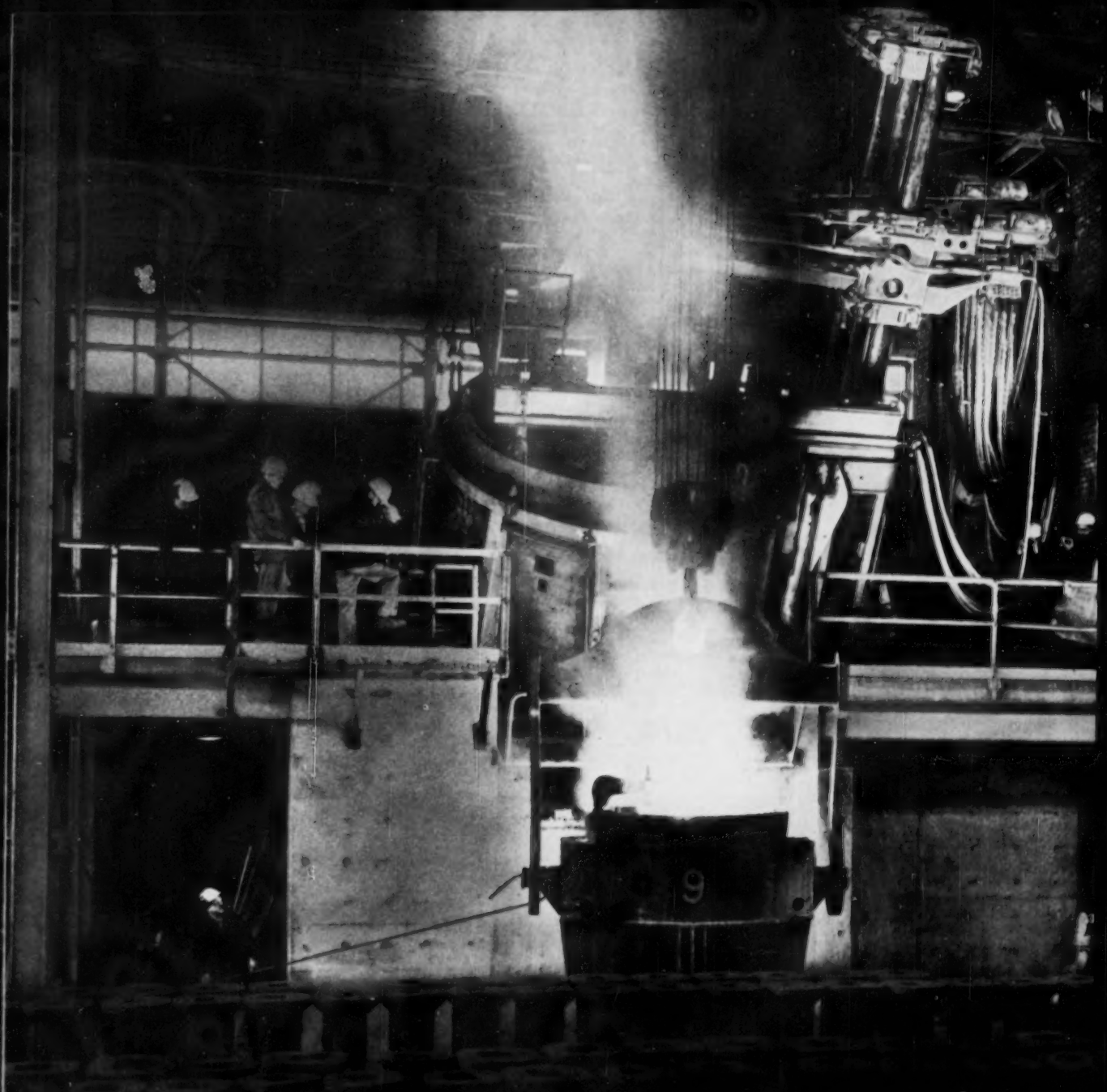
Assistant Plant Metallurgist or Mill Foreman, under 50. Min Dr or related field desired, but not mandatory. To supervise operation; should have knowledge of crushing, grinding, cycloning, flotation, filtering, pumping and drying operations and equipment. Interest in flotation research desirable but secondary to operations. Will have complete and independent charge of operations of present plant upon completion of new plant under construction. For producer of phosphate rock in western U.S. $7200 to start. Sj-5383.

Sales, MetE or equivalent, 30-50. Minimum five to ten years foundry or engineering sales experience relating to metallurgy. Will provide technical sales assistance on ferrous castings. For a ferrous foundry. $8400-$9600, depending on qualifications. Los Angeles area. Sj-5357.

Plant Engineering and Production, EE, MetE, 30-50. a) One mechanical engineer. b) One electrical engineer familiar with heavy electrical usage. c) One metallurgical engineer for production control. They must have seven to twenty years working experience and be familiar with equipment and work in an ore reduction and metals processing plant. Salary commensurate with experience. Nevada. Sj-5349.

Address replies for positions to the nearest local office of the Engineering Societies Personnel Service, Inc., *not* to the JOURNAL OF METALS. Offices are located at 29 E. Madison St., Chicago 1, Ill. . . . 57 Post St., San Francisco, Calif. . . . and 8 W. 40th St., New York 18, N. Y.

A weekly bulletin of engineering positions open is available to AIME members at a subscription rate of $3.50 per quarter or $12 per annum; $4.50 per quarter or $14 per annum for nonmembers, payable in advance. Local offices of the Personnel Service are at 8 W. 40th St., New York 18; 57 Post St., San Francisco; and 29 E. Madison St., Chicago 1.



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Progress in Semiconductors, vol. 4, edited by A. F. Gibson, et al, *John Wiley & Sons Inc.*, 291 pp., $10.50, 1960—This fourth volume in the series contains papers from the USSR, UK, Germany, the Netherlands, Japan, and the US. The following are the titles of papers included: *Negative Effective Masses in Semiconductors; Oxidation Phenomena on Germanium Surfaces; Theory of Avalanche Multiplication in Non-polar Semiconductors; Internal Field Emission; Noise in Semiconductors; The Electrical Effects of Dislocations in Semiconductors; Dielectric Properties of Solids in Relation to Imperfections; and Non-Crystalline, Amorphous, and Liquid Electronics Semiconductors.* •

Les Traceurs Radioactifs en Metallurgie Physique, by C. Leymonie, *Dunod*, Paris, 221 pp., approx. $6.40, 1960—The first chapter is a refresher on radioactivity, discussing atomic structure, isotopes, radioactive decay, and the production of artificial radioisotopes, summarizing their use in metallurgy. The remainder of the book discusses in detail such metallurgical phenomena as diffusion, segregation, corrosion, and oxidation, showing the radioactive tracers in detective operations. Particular attention is given to critical examination and comparison of English, German, and Russian detection methods, especially in ferrous metallurgy. •

Materials and Techniques for Electron Tubes, by W. H. Kohl, *Reinhold Publishing Corp.*, 638 pp., $16.50, 1960—This updated version of material published in 1957 covers the composition, properties, and behavior of materials used in electron tubes, as well as the techniques for assembling them. The materials discussed include glass, ceramics, mica, carbon, and graphite, base metals, and rarer metals. Assembly techniques covered include metals brazing and glass-metal and ceramic-metal sealing. •

Symposium on Radioisotopes in Metals Analysis and Testing, ASTM Special Technical Publication No. 261, 62 pp., $2.75, 1960—The first four papers are of the survey type, covering general types of analysis using radioisotopes, such as thickness gaging, absorptiometry, radiometry, activation and isotope dilution assays, and the proper instrumentation for these methods. The remaining four papers describe current industrial activity, including the anaylsis of tungsten and aluminum for trace impurities, and the training of personnel in the use of radioisotopes. •

International Symposium on High Temperature Technology—Proceedings, *McGraw-Hill Book Co. Inc.*, 348 pp., $15, 1960—These papers are derived from a Stanford Research Institute symposium on fundamental research on high-temperature technology in the US and abroad. There is a section on techniques and measurements which comprises papers on the image furnace . . . on measurement of temperatures and properties . . . and on reaction. Metals, oxides, refractories, and thermal transfer are dealt with in the materials section. Processes discussed are high-pressure methods, condensed-state reactions, fused-salt chemistry, pyrometallurgy, and high-temperature chemical synthesis. Other topics covered are mechanical properties of metals, thermoelectric power, mass spectrometry in high-temperature chemistry. •

Vacuum Processing in Metalworking, by J. W. Cable, *Reinhold Publishing Corp.*, 202 pp., $5.50, 1960—This is a description rather than rigidly technical presentation of vacuum processing. The first three chapters cover

(Continued on page 654)

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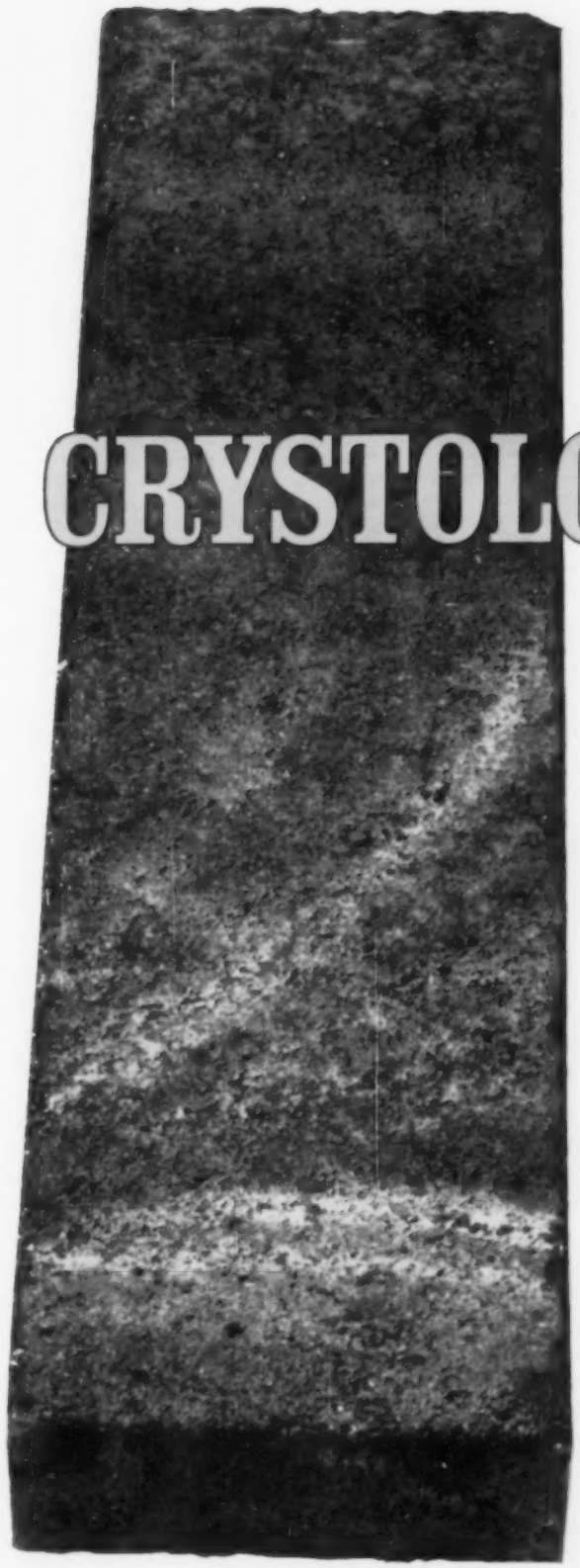

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BOOKS

(Continued from page 652)

vacuum's history . . . production of vacuum . . . and heating systems for vacuum processes. The remainder of the book describes vacuum techniques for refining, purification, melting, casting, plating, brazing, welding, and heat treatment of metals, and for sintering in powder metallurgy. •

Tin and Its Alloys, edited by E. S. Hedges et al, *Edward Arnold Ltd.*, London, 424 pp., $27.50, 1960—Eleven chapters by five British experts on the properties and applications of tin and its alloys describe the tin industry in general . . . the versality, physical, and chemical properties of tin alloys . . . chemical behavior during electrodeposition, hot tinning, and corrosion . . . and practical uses of tin and its alloys. •

Symposium on Electron Metallography, ASTM Special Technical Publication No. 262, 128 pp., $4.25, 1960—This volume includes papers on the electron metallography of cast nickel, tin alloys, and tin plate alloy layers. It also covers dislocations and high-temperature alloys, with special attention to their phase morphology. Other papers discuss electron diffraction techniques and electron microprobe measurements. •

Magnesium and Its Alloys, by C. S. Roberts, *John Wiley & Sons Inc.*, 230 pp., $9, 1960—The extraction and refining of magnesium . . . alloy theory and phenomena . . . and the physical and chemical properties of the metal and its alloy systems are covered in detail. The technology of both cast and wrought alloys is included. •

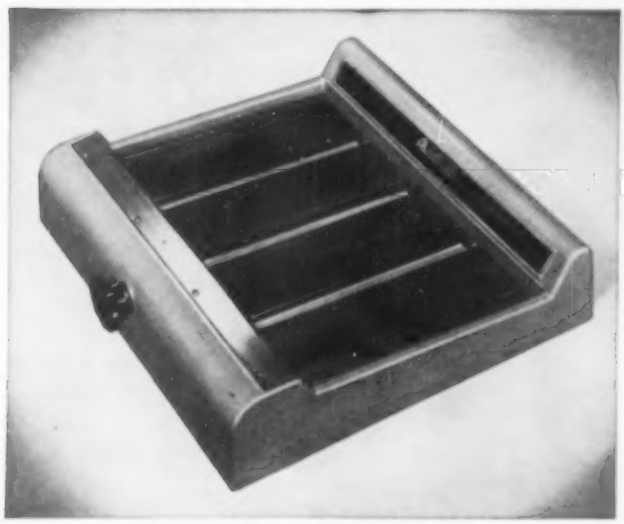
Optics of Thin Films, by A. Vasicek, *Interscience Publishers Inc.*, 403 pp., $12.50, 1960—This is an enlarged and revised edition in English of the author's 1956 Czechoslovakian publication. It reflects his personal concept of the subject and presents those problems which he regards as important in studying thin films; it is a thoretical approach. Included are discussion of reflection and refraction of light by one and more than one thin films, and on a boundary between two and more than two dielectrics. Also discussed are thin metallic films and thin dielectric films on metal, plus the influence of thin films on polarization of light at total reflection. •

Mechanical Properties of Intermetallic Compounds, edited by J. H. Westbrook, *John Wiley & Sons Inc.*, 435 pp., $9.50, 1960—The outgrowth of a 1959 Electromechanical Society symposium, this book includes papers on the mechanical behavior of intermetallics . . . theoretical and experimental investigations of particular properties . . . and experimental techniques for the preparation and study of intermetallics. Appended are an extensive review of the literature in the field, dating from 1908, and an index to the compounds discussed. •

ASTM Standards on Light Metals and Alloys, 5th edition, 355 pp., $4.50, 1959—Includes specifications and general methods of testing cast and wrought aluminum and magnesium alloys . . . methods of testing conductivity and resistivity . . . and some specifications for filler metal and recommended practices for electroplating aluminum alloys. •

Metallizing of Plastics, by Harold Narcus, *Reinhold Publishing Corp.*, 208 pp., $5.50, 1960—A summary of the methods employed by Electrochemical Industries Inc. for metallizing nonconductors, particularly plastics. It deals with actual production procedures, formulations, and techniques. Included are descriptions of deposition by chemical reduction, vacuum metalizing, the cathode-sputtering process, the silver spray method, and the electrolytic method. Also discussed are the characteristics and testing of metallic deposits, plus the recent developments and potential uses for metallized plastics. •

(Continued on page 658)



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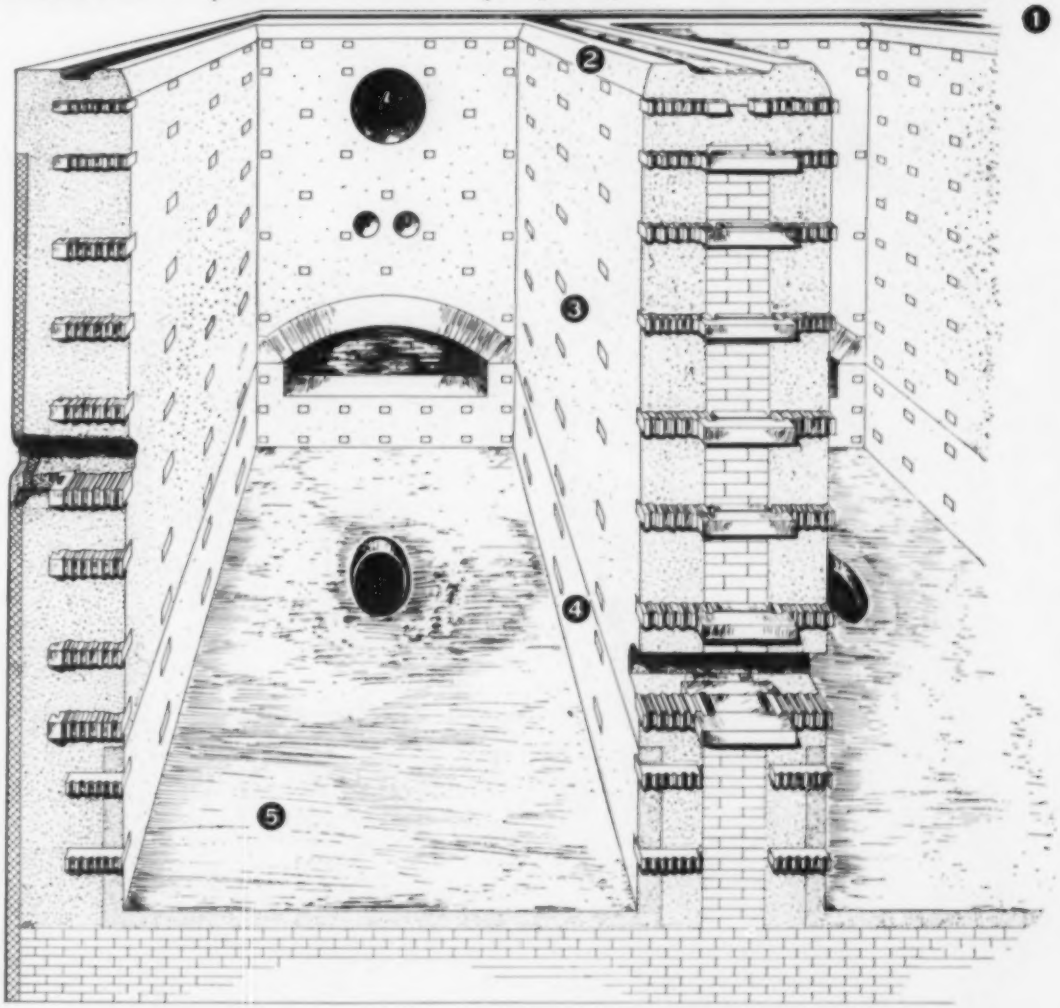

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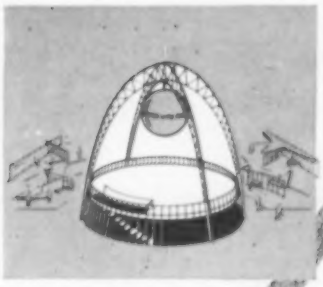

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METALGRAMS

UNION CARBIDE METALS

. . . news about "Electromet" ferroalloys and metals

SEPTEMBER 1960

MORE ELECTROLYTIC MANGANESE -- To meet the increasing demand for "Elmang" electrolytic manganese metal, Union Carbide Metals has expanded its cell capacity by 20 per cent. Hence, a larger supply of this high-purity product (99.9 per cent minimum manganese) is assured for additions to steel, copper, and aluminum. Also of interest: (1) The metal is now available in 50-lb. bags for convenient, accurate additions. (2) The 600-lb. drums of "Elmang" manganese are now color-coded to allow easy identification of the regular, dehydrogenated, and nitrogen-bearing grades. Write for new specification sheet, F-20,151.

* * *

ALLOYS FOR BASIC-OXYGEN STEELMAKING -- Interest has been focused on the basic-oxygen process because it can make high-quality steel rapidly and at low cost. Small accurate alloy additions can be made to the ladle of basic-oxygen heats with bagged alloys. Where large chromium and manganese additions are required, "Chromtemp" ferrochrome and "Mantemp" ferromanganese can be added to the ladle. All these unit-weight packages allow precise analysis control. Also, furnace time is not required to dissolve the alloys in steel. Your UCM representative will be glad to give you further details. Also, write for the article, "O_2 Steelmaking," in the Spring issue of UNION CARBIDE METALS REVIEW.

* * *

SILICON IN COPPER -- First developed in France in 1881, silicon bronze is growing rapidly as a corrosion-resistant material for vessels, tubing, marine and poleline hardware, and electrical conduit. Up to 3 per cent silicon provides strength and hardness. For such additions, Union Carbide Metals offers a low-aluminum silicon metal. Since aluminum makes bronze sluggish in pouring, this special grade contains a maximum of 0.10 per cent aluminum. For further information, contact your UCM representative. Also, write for the article, "Silicon Strengthens Copper," in the Winter 1960 issue of UNION CARBIDE METALS REVIEW.

* * *

SERVICE ON THE MOVE -- Every year, experienced metallurgists of UCM's Metallurgical Service Division travel tens of thousands of miles to customers' plants. Their mission: to provide on-the-site help on the use of ferroalloys and alloying metals in melting. As an example, a Metallurgical Service representative worked with Electric Steel Foundry Company of Portland in evaluating the new fast-dissolving "Simplex" ferrochrome in stainless steel. The joint effort showed that 5- to 7-minute savings in furnace time could be made per 2,000 lb. heat. For a picture story of this cooperative service, write for "Service on the Move" in the Winter 1960 issue of UNION CARBIDE METALS REVIEW.

* * *

HIGHER DUCTILITY WITH CALCIUM-SILICON -- Two Midwestern steel foundries recently obtained data on Grade B steel castings made with and without calcium-silicon. In one test of 91 production heats, all of the heats treated with calcium-silicon and aluminum produced castings with satisfactory ductility. As a comparison, over 25 per cent of the heats deoxidized only with aluminum failed to meet reduction-in-area specifications. In another test of 20 production heats, a 15 to 20 per cent improvement in ductility (% RA) was reported. Further data can be obtained by writing for the article, "Calcium Improves Ductility of Steel Castings," in the Summer 1960 issue of UNION CARBIDE METALS REVIEW.

* * *

UNION CARBIDE METALS COMPANY, Division of Union Carbide Corporation, 270 Park Ave., New York 17, N. Y. In Canada: Union Carbide Canada Ltd., Toronto.

"Chromtemp," "Electromet," "Elmang," "Mantemp," "Simplex," and "Union Carbide" are registered trade marks of Union Carbide Corporation.

BOOKS

(Continued from page 654)

Questions of the Metallurgy and Physics of Semiconductors, edited by D. A. Petrov, Morris D. Friedman Inc., 143 pp., no price quoted, 1960—This translation from the Russian includes 19 papers presented during a conference on the subject. They include discussion of current problems . . methods of obtaining high-purity silicon and single-crystal silicon with given electrical characteristics . . . chemical-compound semiconductors . . . alloys of bismuth-technetium and cobalt-antimony systems . . . vitreous semiconductors . . . and impurities and etches of germanium and silicon. •

Design Information on 5Cr Alloy Steels for Aircraft and Missiles, by R. J. Favor and W. P. Achbach, Defense Metals Information Center, Battelle Memorial Institute, 48 pp., $1.25, 1960—This volume presents metallurgical and manufacturing practices, and mechanical and physical properties of the forenamed steels. Elevated-temperature design curves on short-time ultimate tensile strength, tensile yield strength, comprehensive yield strength, ultimate shear strength, bearing ultimate strength, and bearing yield strength are given. Typical stress-strain data are summarized.

Order from the *Office of Technical Services, Dept. of Defense, Washington 25, D. C.*, using the code number PB 151072.

Physical Metallurgy of Precipitation-Hardenable Stainless Steels, by D. C. Ludwigson and A. M. Hall, Defense Metals Information Center, Battelle Memorial Institute, 73 pp., $2, 1960—Three classes of steels—martensitic types, semi-austenitic types, and austenitic types—are separately treated herein. The present knowledge of the physical metallurgy of precipitation-hardenable stainless steels is summarized. In addition, the manner in which hardening mechanisms govern heat treatment and fabrication is discussed. Approximate mechanical properties are presented for illustration. A review also is given of the classification of stainless steels.

Order PB 151067 from the *Office of Technical Services, Dept. of Defense, Washington 25, D. C.*

The Structure of Aluminum-Silicon Alloys, by R. A. Meussner, US Naval Research Laboratory, 25 pp., $0.75, 1960—Morphologies of the normal (unmodified) aluminum-silicon alloys were examined by a variety of techniques. A chemical extraction process provided specimens of primary and eutectic silicon particles, as well as primary alpha dendrites for visual and X-ray examination. Adaptation of two etching procedures permitted detailed study of the eutectic structure and growth characteristics of individual phases. Correlation of these microstructural characteristics with the forms of the extracted phase particles was facilitated by exposing the silicon phase in the microstructure to a deep electrolytic etch. Although the dendritic form of the primary alpha phase was found to be unaltered by changes in growth conditions, the form of the primary silicon phase was affected. The usual complex idiomorphic form of the silicon phase was transformed to the dendritic form when growth occurred in super-saturated liquid. Eutectic silicon plates dominate the eutectic transformation and, by branching, restrict growth of the eutectic alpha phase. This method of solidification produces fine eutectic

(Continued on page 670)

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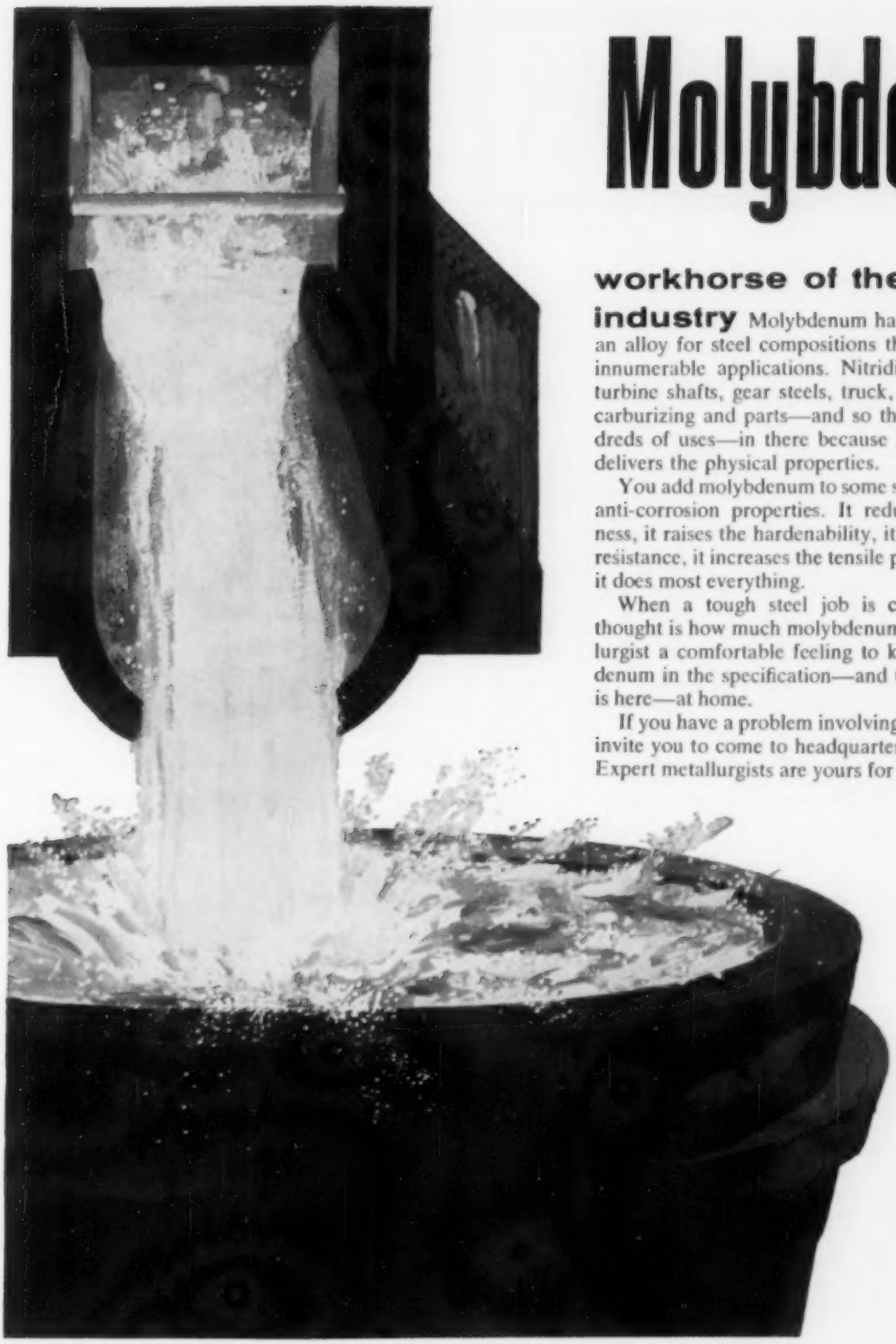

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TRANSACTIONS

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Recently Accepted TRANSACTIONS Papers

The Reduction of the Iron Values of Ilmenite to Metallic Iron at Less than Slagging Temperatures, by R. H. Walsh, H. W. Hockin, D. R. Brandt, P. L. Dietz, and P. R. Girardot (ISD)

The Preparation and Properties of Barium, Barium Telluride and Barium Selenide, by Edward Miller, Kurt Komarek and Irving Cadoff (EMD-IMD)

The Effect of Phosphorus on the Deformation and Fracture Characteristics of Iron from 1600° to 2200°F, by Rolf Nordheim, Thomas B. King, and Nicholas J. Grant (ISD)

Hardening of Internal Oxidation as a Function of Velocity of the Oxidation Boundary, by J. L. Meijering (IMD)

Surface Tension and Contact Angles of Copper-Nickel Alloys on Titanium Carbide, by T. J. Whalen and M. Humenik, Jr. (IMD)

Self-Diffusion in Alpha Iron, by Richard J. Borg and C. Ernest Birchenall (IMD)

Elevated Temperature Properties of Lithium Fluoride and Magnesium Oxide Single Crystals, by W. L. Phillips, Jr. (IMD)

Recovery Properties of Lithium Fluoride Single Crystals, by W. L. Phillips, Jr. (IMD)

The Application of Ultrasonic Energy to Ingot Solidification—Part I, by D. H. Lane, J. W. Cunningham, and W. A. Tiller (IMD)

The Application of Ultrasonic Energy to Ingot Solidification—Part II, by D. Lane and W. A. Tiller (IMD)

Some High-Temperature Properties of Copper-Zirconium and Copper-Chromium High-Conductivity Alloys, by Matti J. Saarivirta and Pierre P. Taubenblat (IMD)

Stability Relations of Calcium Ferrites: Phase Equilibria in the System $2CaO \cdot Fe_2O_3$-$FeO \cdot Fe_2O_3$-Fe_2O_3 Above 1135°C, by Bert Phillips and Arnulf Muan (ISD)

Growth of (110) [001] Oriented Grains in High-Purity Silicon Iron—A Unique Form of Secondary Recrystallization, by J. L. Walter and C. G. Dunn (IMD)

The Diffusivity of Hydrogen in α-Iron, by E. W. Johnson and M. L. Hill (IMD)

A Further Study of Ti_2Ni-Type Phases Containing Titanium, Zirconium or Hafnium, by M. V. Nevitt, J. W. Downey, and R. A. Morris (IMD)

Intragranular Precipitation of Intermetallic Compounds in Complex Austenitic Alloys, by H. J. Beattie, Jr. and W. C. Hagel (IMD)

The Solubility of Nitrogen in Liquid Fe-Cr-Ni Alloys, by J. Humbert and J. F. Elliott (ISD)

Solubility of Nitrogen in Liquid Iron Alloys I. Thermodynamics, by Robert D. Pehlke and John F. Elliott (ISD)

Solubility of Nitrogen in Molten Iron-Manganese Alloys, by Sylvan Z. Beer (ISD)

Recrystallization of Cold Drawn Sintered Aluminum Powder, by E. J. Westerman and F. V. Lenel (IMD)

Diffusion in the Iron-Chromium System, by H. W. Paxton and T. Kunitake (IMD)

Resistance Apparatus for the Continuous Measurement of Fine Wire Uniformity, by A. I. Michaels, T. L. Weaver, and R. C. Nelson (IMD)

Purification Reactions of Tantalum During Vacuum Sintering, by W. D. Klopp, D. J. Maykuth, H. R. Ogden, and R. I. Jaffee (EMD)

Self-Diffusion of Copper in Molten Copper, by John Henderson and Ling Yang (EMD)

Self-Diffusion of Aluminum in CaO-SiO_2-Al_2O_3 Melts, by John Henderson, Ling Yang, and G. Derge (ISD)

End-Point Temperature Control of the Basic Oxygen Furnace, by W. J. Slatosky (ISD)

Precision Lattice Parameter Determinations of Zirconium-Oxygen Solid Solutions, by Barry D. Lichter (IMD)

Investigation of Bessemer Converter Smoke Control, by A. R. Orban, J. D. Hummell and R. B. Engdahl (ISD)

Strain Aging in Ag Base Al Alloys, by A. A. Hendrickson and M. E. Fine (IMD)

A Metallographic Study of Precipitation of Copper from Alpha Iron, by E. Hornbogen and R. C. Glenn (IMD)

The Aging Characteristics of Cu-Ti, by G. T. Murray (IMD)

Room Temperature Deformation and Fracture Characteristics of Lithium Fluoride Single Crystals, by W. L. Phillips, Jr. (IMD)

The Effect of Dilute Transition Element Additions on the Recrystallization of Iron, by E. P. Abrahamson, II and B. S. Blakeney, Jr. (IMD)

The Use of Heat- and Mass-Transfer Model Studies in the Evaluation of the Rates of Deposition of Metals in Complex Systems, by G. H. Kesler, J. H. Oxley, and C. E. Dryden (EMD)

The Iron-Carbon-Sulfur System from 1149°C to 1427°C, by Albert M. Barloga, Keith R. Bock, and Norman Parlee (ISD)

Heterogeneous Nucleation in the Liquid-to-Solid Transformation in Alloys, by B. E. Sundquist and L. F. Mondolfo (IMD)

Effect of Structure and Purity on the Mechanical Properties of Columbium, by A. L. Mincher and W. F. Sheely (IMD)

Thermal Diffusivity of Armco Iron, by G. D. Cody, B. Abeles, and D. S. Beers (IMD)

personals

Richard A. Swalin has been promoted from associate professor to professor in the University of Minnesota's Department of Metallurgy. He also has been appointed a director of Baker Engineering Corp., Minneapolis.

Clyde B. Jenni has been selected to deliver the Steel Founders' Society of America's exchange lecture before the British Steel Castings Research Association in England this fall.

Mr. Jenni is director of research and chief metallurgist at the Eddystone, Pa., plant of General Steel Castings Corp. He also is 1960-1961 Chairman of the Eastern section, National Open Hearth Steel Committee. Mr. Jenni received the SFSA's Technical and Operating Medal this year for his contributions to the steel castings industry. The title of the lecture, to be delivered in Harrogate, England, Oct. 20th, will be *Technical Control in Steel Foundries in North America.*

Lars Ekholm is now vice president of Eastern operations for the Climax Molybdenum Co., div. of American Metal Climax Inc. He will be responsible for the production of molybdenum products. Mr. Ekholm formerly was general sales manager of Climax Molybdenum Co.

John Zotos has been appointed assistant professor in the Department of Mechanical Engineering, Northeastern University (Boston). He previously was assistant chief of the Watertown Arsenal Rodman Laboratory's Experimental Foundry Branch. Mr. Zotos is on the Iron and Steel Division's Physical Chemistry of Steelmaking Committee.

Bernard R. Queneau has received an award of nearly $2000 for his part in a process for nucleating steel ingots, now patented by U.S. Steel Corp. Sharing in the award with a similar check was **Dennis J. Carney.**

Dr. Queneau is assistant manager of metallurgy—inspection and research, U.S. Steel's Tennessee Coal & Iron div. Dr. Carney is assistant general superintendent, Homestead District works, Homestead, Pa.

P. L. Schereschewsky is in Singapore on a United Nations Technical Assistance Organization assignment to investigate the feasibility of establishing an iron and steel industry there. Mr. Schereschewsky is a representative of Public Utilities of France, based in Washington, D.C.

William P. Bittenbender is now director of international operations for the Sun Chemical Corp.

Davis S. Fields, Jr., has joined the Department of Mining and Metallurgical Engineering, University of Kentucky. Dr. Fields formerly was with the Alcoa Research Laboratories.

William O. Harms, formerly associate professor, University of Tennessee, has been appointed to the staff of the Oak Ridge National Laboratory.

Alexander L. Feild, Jr., is now a senior engineer in the Astronuclear Laboratory, Westinghouse Electric Corp., Pittsburgh. Mr. Feild previously was a research associate for Universal-Cyclops Steel Corp.

George Havas has been appointed vice president and director of engineering, Kaiser Industries Corp. He has been associated with Kaiser companies since 1928; his last previous position was as general manager of the Henry J. Kaiser Co.'s Heavy Construction div.

Roy S. Jamison has gone to Norwalk Powdered Metals Inc. as a vice president. He was sales engineering manager of the Hoeganaes Sponge Iron Corp.

Fred B. O'Mara is now vice president—production, National Carbon Co., div. of Union Carbide Corp. He had been director of manufacturing for National Carbon since early this year.

Oscar Pearson has become executive vice president of U.S. Steel's Tennessee Coal & Iron div. His most recent post was that of administrative vice president—steel producing divisions.

Roydon S. Pratt has been named technical and research director, Bridgeport Brass Co. With Bridgeport Brass since 1920, Mr. Pratt most recently held the title of staff technical director.

Lowell Steinbrenner is now a development engineer for the Youngstown and Louisville, Ohio, plants of Jones & Laughlin Steel Corp.'s Stainless and Strip div. He formerly was a research engineer in the Stainless Steel section, J&L's Graham Research Laboratories.

Roger M. Wolcott also is involved in a J&L change. He becomes director of production planning programs for the Corp. He will coordinate J&L programs in production planning order processing, and inventory control.

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Extractive and Physical Metallurgy of Plutonium and its Alloys

The Proceedings of a Technical Symposium sponsored by the Nuclear Metallurgy Committee of the Institute of Metals Division, and the Titanium, Uranium, and Uncommon Metals Committee of the Extractive Metallurgy Division, The Metallurgical Society of AIME. San Francisco, February 16-17, 1959.

Edited by W. D. WILKINSON,
Argonne National Laboratory

The addition to these Proceedings of an introductory chapter on the metallurgy of plutonium and of an extensive annotated bibliography has made this volume of even greater value to metallurgists working in this particularly important field. Mr. Wilkinson both edited the Proceedings and provided the new material.

CONTENTS

Introduction to Plutonium Metallurgy

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- Alternative Routes for the Conversion of Plutonium Salts to Metal and Their Recovery Problems
- Calcium Reduction of Plutonium Halides to Metal
- Preparation of Metallic Plutonium
- Distribution of Plutonium and Selected Impurity Elements between Nitrate Solutions and Tri-n-butyl Phosphate
- The Preparation of Plutonium Halides for Fused Salt Studies
- Removal of Fission Product Elements from Plutonium by Liquidation

PHYSICAL METALLURGY

- Some Principles of the Alloying Behavior of Plutonium
- The Plutonium-Cerium Diagram
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- Metallography of Alpha Plutonium
- The Etching of Plutonium and Its Alloys by Cathodic Bombardment
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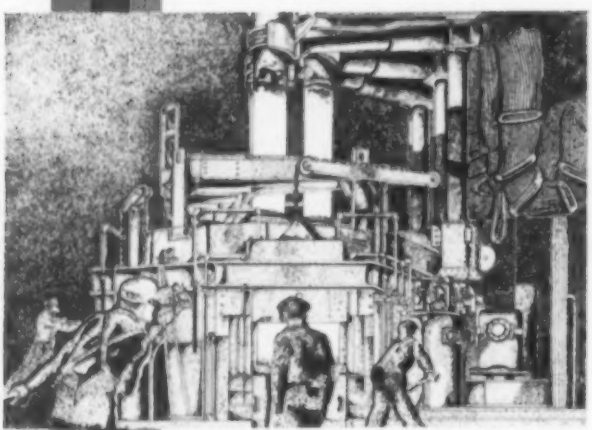

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New nozzle technique developed

Plasmadyne Corp., Santa Ana, Calif. and Electroform Corp. of Los Angeles have developed a new technique which allows production quantities of rocket nozzles with throat dimensions to extremely close tolerances at considerable cost reduction.

The lining of the nozzle is plasma jet sprayed onto a rapidly spinning mandrel manufactured to the desired tolerance. Once the lining is deposited to the desired thickness, back-up layers of support metals can be electroformed to the liner and the mandrel can then be leached out of the finished nozzle.

The first test nozzle produced was formed on a mandrel sprayed with a 0.030 in. thick lining of tungsten followed by a 0.100 in. layer of electrodeposited nickel onto the liner to act as support; and then chrome flashed the exterior. The mandrel was etched out with a solution of sodium hydroxide.

Larger Mo ingots

Refractomet div. of Universal-Cyclops Steel Corp., Bridgeville, Pa., has announced successful melting of a large arc-cast molybdenum ingot. The ingot, 17 in. in diam and 69 in. in length, weighed 5,000 lb. A significant increase in the size of finished forgings will be possible with the larger forging billet diameters.

Climax Molybdenum Co., a division of American Metal Climax Inc., is producing ingots containing 85 pct W and 15 pct Mo in sizes up to 11 in. in diam. The product is reportedly the only full-density alloy available in such large sizes that will withstand temperatures over 5500°F.

England's first LD Steel

Britain's first steelmaking converter to use the LD process is in full three-shift operation at the Ebbw Vale works of Richard Thomas & Baldwins. The vessel has a capacity of 30 tons, and includes provision for the injection of powered lime through the oxygen lance.

Electron beam welding Mo and W

Hamilton Standard, a division of United Aircraft Corp. has been awarded an Air Force contract to study welding of molybdenum and tungsten by electron beam. Hamilton Standard holds North American rights to electron-beam welding and cutting equipment developed by the Carl Zeiss Foundation of West Germany.

Both molybdenum and tungsten are difficult to weld by normal techniques. Under high welding heats—5000° to 6000°F—they tend to become brittle. Unless heat is confined to the immediate weld, which is almost impossible with conventional techniques, there is serious loss of the metals strength.

In the Hamilton-Zeiss machine, the electron welding beam can be focused on a spot less than 0.001 in. in diam. Hamilton will test butt, lap, seam, edge, and spot welds in sheets of tungsten and molybdenum ranging in thickness from 0.005 to 0.1 in.

Aluminium develops new process

Aluminium Limited plans the construction of new facilities at Arvida, Que., Canada, for the production of aluminum by a basically new process, Nathanael V. Davis, president, has announced.

Mr. Davis said, "The process has been under development for some years through the laboratory and pilot plant stages. It employs, as its principal raw material, bauxite of roughly similar grades to that required by the conventional process. The new process does not appreciably reduce the large power requirement which is an outstanding characteristic of the conventional process. It does, however, give promise of substantial savings in other elements of production cost and in the investment required per ton of capacity."

Unofficial sources, with no confirmation by the company, suggest that the Canadian company will use a production method that will convert bauxite directly into aluminum—bypassing the alumina stage altogether. This would represent the first revolutionary development in aluminum since perfection of the Hall process.

The new facilities, designed as an experiment unit, will have a capacity of 6000 to 8000 tons of aluminum per year and are expected to be in operation within two years. The new plant will cost about $4 million.

Atlas tests uranium-steel

Atlas Steels Ltd., Welland, Ont., Canada has been test-rolling heavy ingots of uranium-steel alloy. Reportedly, the plate confirms earlier tests made of the alloy's two properties: corrosion and fatigue resistance.

The Canadian federal mines laboratories inaugurated the test on the theory that uranium might be used to improve steel's qualities, as well as provide a market for Canada's abundant uranium supplies.

Initial experiments involved the use of from 1 to 4 lb of uranium per ton of steel.

Union Carbide studies alloys

Union Carbide Metals Co., in cooperation with the Air Force, is undertaking a program to accumulate the necessary background information on refractory metals for the development of a columbium-tungsten-tantalum alloy applicable at temperatures up to 3500°F. The program will be carried out in three phases covering the effect of interstitials, strengthening mechanisms, and oxidation characteristics.

The first phase will concern the effect of interstitial impurity elements on mechanical properties of refractory metals as a class; the second phase will cover high-temperature strengthening mechanisms for refractory metals including alloying, dispersion hardening, and solid solution strengthening; and the third phase will be a study of oxidation behavior in the temperature range of 2000° to 3000°F. Particular attention will be directed to the columbium- and tantalum-base alloys which show maximum oxidation resistance.

REPORTER

Vanadium-Columbium alloys studied

Vanadium-base alloys containing 20 to 50 pct Cb display good high-temperature strength, good corrosion-resistance in both oxidizing and reducing environments, and fabricability by conventional hot-working techniques in air, according to a recent report by Dr. S. T. Wlodek, research metallurgist at the laboratories of Union Carbide Metals Co.

He found that in the warm-worked condition vanadium-columbium alloys have ultimate tensile strengths of 120,000 to 35,000 psi over the temperature range of 700° to 1000°C and stress-rupture properties at 700°C corresponding to 100-hr life at stresses in excess of 100,000 psi.

Both the aqueous corrosion resistance and oxidation resistance of the alloys were also considerably higher than those of pure vanadium. Maximum oxidation resistance is obtained by titanium and aluminum additions.

OLP process in Europe

The OLP process for top blowing with oxygen and powdered lime is now under intensive development in Europe. At Dillingen in the Saar, a 30-ton converter is in industrial use on high-phosphorous iron. At Usinor's plant in Denain in France, a 60-ton OLP converter is starting in the autumn, and a second one of the same capacity will start at the end of the year. The Dunkirk plant, now under construction, will also use the OLP on its 140-ton converter. The process is described in detail in the July 1958 issue of JOURNAL OF METALS.

New generator for satellites

A compact, lightweight generator which can convert heat from cerium-144, a by-product of nuclear reactors, into electricity, is being prepared for testing by the Martin Co. of Baltimore. The generator will power advanced satellites and space probes for at least a year.

The generator's outer shell is aluminum with an inner liner of stainless steel. The core is built mainly of Inconel X. In the tests, the generator will contain 4000 lb of mercury, which would be omitted in space operation, bringing the flight weight down to about 175 lb. The fuel capsule, containing tightly-sealed pellets of radioactive cerium-144, is located at the center of the generator, supported by light tubing.

Roughly egg-shaped, the generator is 34 in. long and 24 in. in diam. Most of its surface is dotted with screw headed caps marking the location of 277 thermocouples. These thermocouples are connected in series to add up to 125 w at 28 v.

USBM announces new program

As part of a continuing cooperative project with the Atomic Energy Commission, scientists of the Bureau of Mines will seek uses for depleted uranium, material from which most of the fissionable isotope, U-235, has been extracted. Research at the Rolla, Mo., Metallurgy Research Center has shown that depleted uranium added to steel makes a high-strength alloy and that it also can improve the quality of bearing metals. Technologists at Bruceton, Pa. are working to develop a depleted uranium catalyst that can minimize atmospheric pollution from automobile exhaust gases.

Bureau metallurgists at Salt Lake City will experiment with combined chemical and physical upgrading processes for low-grade and complex uranium ores.

Studies to develop techniques and methods for electrowinning and electrorefining of uranium metal will be continued at the Bureau's Reno, Nev., Metallurgy Research Center and at its Boulder City, Nev., Metallurgy Research Laboratory.

Germanium: The Department of Interior has announced that the Bureau of Mines has begun a research program on germanium, one of the major semiconductor materials of the electronics industry. The program will include the development of simpler methods for detecting germanium and investigations of the economic recovery of germanium from coal fly-ash. Although germanium is a minor constituent of coal, efforts to recover germanium in commercial quantities from this source have not been successful. In the US, germanium is obtained solely as a byproduct of base-metal processing.

The Bureau's program was prompted by the growing dependence on foreign sources and the increased use of germanium. The research program will be carried out at the Bureau's Rolla, Mo., and Bartlesville, Okla., installations.

Rare-earths: Research projects in the field of rare-earth metals will include extraction and separation, physical metallurgy, and fused-salt electrowinning of the light and heavy sub-groups of the rare-earth elements. Spectrochemical, fluorescent X-ray spectrography, and radio-chemical techniques will have roles in the research.

Projects at the Bureau's Metallurgy Research Center, Reno, Nev., will include physical processing methods, solvent extraction, ion exchange, and other chemical methods of recovering rare-earth elements and yttrium from ores, concentrates, plant residues, and wastes. The Bureau will also emphasize the fused-salt electrowinning method in search of continuous production of higher purity rare-earth metals.

Tellurium: A promising metal for the field of thermoelectricity, tellurium is the object of intensified research now under way at the Bureau's installations in Salt Lake City, Utah, and Spokane, Wash. The program includes a wide search for new domestic sources of tellurium.

Boron preparation: Old and new techniques for preparing boron will be investigated at the Metallurgy Research Center in Albany, Ore. Reduction of boron halides with active metals and also the electrolysis of baths containing boron halides in various organic solvents will be studied.

Research on purification and processing will cover sintering boron samples in vacuum induction furnaces, electric-arc melting of boron in vacuum or inert atmospheres, electron bombardment, and new and promising purification techniques.

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education

LITERATURE

► *Concise Iron and Steel Dictionary* is the title of a 110-pp. English-Russian, Russian-English reference work available free. In addition to covering the main iron and steel-making expressions, particular attention is paid to the terms connected with the continuous casting of steel.

Besides the vocabulary itself, there is a brief history in English of the development of the continuous casting process. A chapter in Russian reviews developments in the UK since the process was first patented. Maps of the UK and of the USSR show the location of continuous casting plants in relation to the rest of the industry and its raw materials resources. Diagrams with bilingual text illustrate the actual process and plant.

Write the Information Office, British Iron and Steel Federation, Steel House, Tothill Street, London, S.W.1.

► The Carborundum Metals Co.'s Zirconium Information Center is offering a free *Zirconium Data File*. It gives charts, pictures, tables, and graphs on production of zirconium and hafnium; wrought products; corrosion-resistant applications; fabrication; electroplating; powder metallurgy; etc.

For copies, write the Zirconium Information Center, Carborundum Metals Co., Akron, N. Y.

► Speer Carbon Co. is giving away an 8-pp. booklet on carbon and graphite products. The booklet includes data on the mechanical, chemical, and electrical properties of carbon and graphite. Charts, graphs, dimensional diagrams, and illustrations are included.

Write Merle Lecker, Speer Carbon Co., St. Marys, Pa.

► A revised edition of *Recommended Practices for Metallizing Shafts or Similar Objects* has been published by the American Welding Society.

A new section has been introduced which deals with Surface Preparation for Metallizing. Most of the other material has been up-dated.

Copies of the 32-pp. booklet are available for $1 from the American Welding Society, Dept. 7, 33 West 39th St., New York 18, N. Y.

Wiley BOOKS

MAGNESIUM AND ITS ALLOYS

By C. Sheldon Roberts, *Fairchild Semiconductor Corp.* A balanced picture of scientific principles and technological know-how involved in the fabrication and use of magnesium. Covers extraction, refining, casting, and chemical and physical properties. *One of the Wiley Series on the Science and Technology of Materials, J. H. Hollomon, Advisory Editor. 1960. 230 pages. $9.00*

RADIATION PYROMETRY AND ITS UNDERLYING PRINCIPLES OF RADIANT HEAT TRANSFER

By T. R. Harrison, *Brown Instruments Div., Minneapolis-Honeywell Regulator Co.* Covers three major areas: 1) theory; 2) principles; 3) details on construction and operation of instruments. *1960. 234 pages. $12.00*

NON-CRYSTALLINE SOLIDS

Edited by V. D. Frechette, *State University of New York,* with an Editorial Committee of five. Records results of a conference on current progress in defining and identifying amorphous structures, and assessing methods of investigation and results so far obtained. *1960. 536 pages. $15.00*

THERMOELECTRICITY

Edited by Paul H. Egli, *U. S. Naval Research Laboratory.* Four sections cover: a broad survey of basic concepts; the physics of thermoelectric performance; important problems in thermoelectric research; and the relative merits of static and transient methods of measuring thermal conductivity. Includes a synthesis by Dr. Egli of the ideas in the main body of the book. *1960. Approx. 416 pages. Prob. $10.00*

SURFACE EFFECTS ON SPACECRAFT MATERIALS

Edited by F. J. Clauss, *Lockheed Aircraft Corp.* Records a symposium sponsored by Lockheed and the U. S. Air Force, in 1959. It includes methods for calculating required characteristics, available materials, laboratory techniques for measuring radiation characteristics, and effects of space environment on behavior of materials. *1960. 404 pages. $11.50*

DYNAMIC BEHAVIOR OF THERMOELECTRIC DEVICES

By Paul E. Gray, *I.T.* Explores small-signal dynamic behavior of thermoelectric heat pumps and generators. This approach permits analysis of linear models describing behavior of these devices. Then, these analyses give a number of small-signal transfer functions for use in computing response of devices in either freqency- or time-domain. *A Technology Press Research Monograph, M.I.T. 1960. 136 pages. $3.50*

PROPERTIES AND STRUCTURE OF POLYMERS

Elucidated by Stress Relaxation Studies.

By Arthur V. Tobolsky, *Princeton University.* Reduces the seemingly complex physical properties of polymers to easily-understood generalizations. It also shows how the study of mechanical behavior of polymers can be used to solve problems in polymer chemistry. *1960. 331 pages. $14.50*

PROGRESS IN SEMICONDUCTORS, Vol. IV

Edited by A. F. Gibson, R. E. Burgess, *and* F. A. Kroger. *1960. 292 pages. $10.50*

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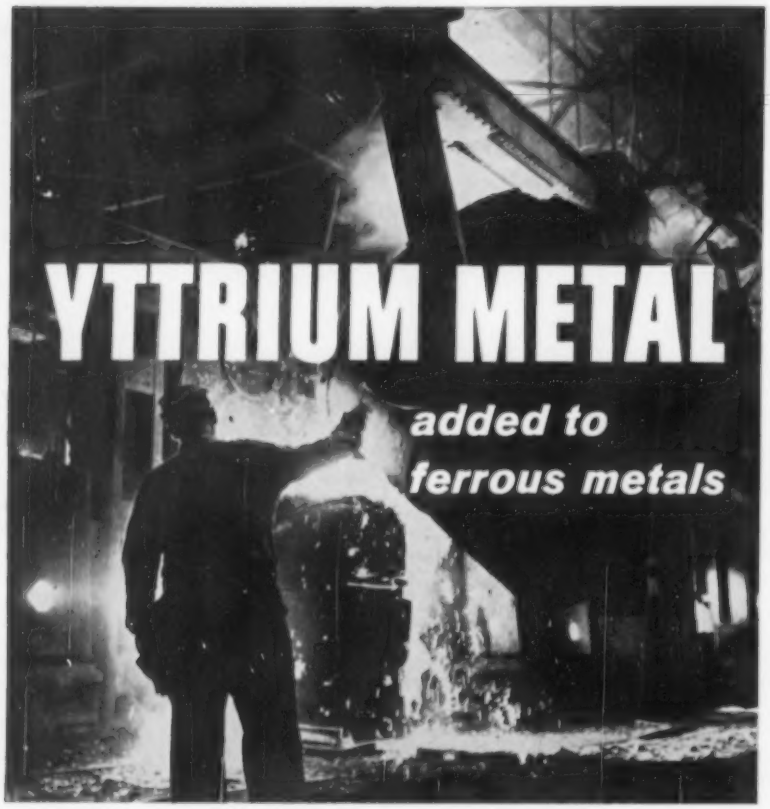

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letters

to the editor

Smelting Wet Concentrates

Subject: *Smelting Practice at Noranda and Gaspe*, by J. N. Anderson, Noranda Mines, Ltd., p. 482, June, Journal of Metals.

I have not seen nor read Jack Anderson's paper, as I live in Arizona.

However, quote: "Although Noranda has set many fine records in the smelting of hot calcines, the newest reverberatory furnace receives wet concentrates." Unquote.

From the way this sentence is phrased, one would think that Noranda men never had designed a reverberatory furnace which would smelt wet concentrates. Just to set the record straight, the San Manuel reverberatory furnace at San Manuel, Ariz., was designed by me and put into operation January 1956—the first reverberatory furnace in the US with magnesite suspended roof for smelting wet copper ores and wet concentrates. It has been running ever since, except when there was a strike, and I believe it has smelted more tons of wet charge per day than any other reverberatory furnace in Canada or the US.

W. B. Boggs
Tucson, Ariz.

[The paper, *Smelting Practice at Noranda and Gaspe*, by J. N. Anderson of Noranda Mines, Ltd., was presented during the International Symposium on the Extractive Metallurgy of Copper, Nickel, and Cobalt—the proceedings of which will be published later this year by Interscience Publishers Inc. **Ed.**]

THE ENGINEERING INDEX

Here is a guide to the current technological literature of the world. More than 1500 bulletins, reports, transactions, and periodicals are reviewed and abstracted to supply engineers with up-to-the-minute developments in their respective fields of interest. Information on a range of 249 subject divisions is classified on 3 x 5 in. cards, then passed on to subscribers to the index service in the form of daily and weekly reports.

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editorial

Into the Season's Meetings

Approaching the beginning of the annual meeting season, we can again look forward to a rich harvest of information-filled meetings provided for The Metallurgical Society members by various hard-working program chairmen.

First to come up is the *Rocky Mountain Minerals Conference* on Oct. 5-7 in Salt Lake City. The conference will offer papers on fused salt electrolysis, reagent system design, rock mechanics, and undersea mining.

Next is the *Fall Meeting* of The Metallurgical Society in Philadelphia Oct. 17-20. One of the features of the meeting will be a symposium on Metallurgy in the Army (see page 708-709).

The AIME *Electric Furnace Conference*, to be held in Chicago Nov. 30-Dec. 2, will offer technical sessions on such subjects as non-metallic inclusions in steel, factors in improving steel quality, some developments in steelmaking processes, and refining metal in air induction melting.

On Jan. 18, 1961, a *Conference on Bar and Shaped Products*, sponsored by the Mechanical Working Committee of The Metallurgical Society will be held in Pittsburgh. At the morning session, papers dealing with surface preparation and rolling of primary products will be presented. The afternoon session will feature the presentation of papers on metallurgical factors in rolling blooms and billets.

The AIME *Annual Meeting* will take place in St. Louis, Feb. 26-Mar. 2. One of the special features of the meeting will be a symposium on Metallurgy in the Air Force. The Howe Memorial Lecture of the Iron and Steel Division will be presented in addition to the annual lectures of the Institute of Metals Division and the Extractive Metallurgy Division.

On Apr. 10-12, 1961, the 44th *National Open Hearth Steel Conference and Blast Furnace, Coke Oven, and Raw Materials Conference* will be held in Philadelphia.

Philadelphia will also be the site of the *International Symposium on Agglomeration* which will be held on Apr. 12-14, 1961. The Symposium will deal with sintering, pelletizing, nodulizing, briquetting, powder metallurgy, ceramic bonding, and related processes.

There are, in addition, a host of local section and regional meetings, too numerous to mention here. We hope to see you at some of them as well as the major national meetings here listed—each will be an outstanding event. WEL

BOOKS

(Continued on page 658)

colonies in this abnormal eutectic structure.

Order PB 151760 from the *Office of Technical Services, Dept. of Defense, Washington 25, D. C.*

Classification of Soviet Gas-Turbine Steels: Review of Soviet Literature 1954-1959, 17 pp., $0.50, 1960—Soviet heat-stable and scale-resistant steels are arranged into seven classes—in order of increasing service temperature limits, based on chemical composition, methods of production, and methods of strengthening.

Order the report, 60-21016, from the *Office of Technical Services, Dept. of Defense, Washington 25, D. C.*

Selected References on Brittle Fracture, by Don Owens and Lynne Doss, Information Management Division, Battelle Memorial Institute, 63 pp., no price quoted, 1960—The purpose of this bibliography is to bring together the results of past and current studies of the subject in view of its renewed importance as an obstacle in the missile and space programs. The following subjects have been categorized and cross referenced: testing techniques . . . low temperature fracture . . . fracture of welded structures . . . influence of processing variables other than welding . . . temper brittleness . . . hydrogen embrittlement . . . influence of composition on brittle fracture . . . ductile-to-brittle transition . . . stress-corrosion cracking . . . fundamental studies and status of research . . . fracture of nonferrous materials . . . design considerations . . . case histories . . . and methods of circumventing brittle fracture.

Order PB 161205 from the *Office of Technical Services, Dept. of Defense, Washington 25, D. C.*

A Summary of Comparative Properties of Air-Melted and Vacuum-Melted Steels and Superalloys, by D. C. Ludwigson and F. R. Morral, Battelle Memorial Institute, 144 pp., no price quoted, 1960—Comparative data presented show that: 1) the reduction in hydrogen content of large heats of low-alloy steels effected through vacuum pouring can lead to improved ductility; 2) the reduction in gas, inclusion, and impurity content of low-alloy or chromium hot-worked die steels that can result from either vacuum-induction melting or consumable-electrode vacuum-arc remelting can lead to improved fatigue and impact properties, higher ductility, and better behavior under multiaxial stresses, but seldom improves tensile strength; 3) the transition temperature, rupture strength, and elongation of ferritic and martensitic stainless steels may be improved by vacuum-induction melting; 4) the improved cleanliness, the better control of composition, and the modifications of composition achieved by vacuum-induction melting have lead to improved stress-rupture, fatigue, and tensile properties in nickel-base superalloys; 5) the tensile and rupture strengths of cobalt-base superalloys are affected but little by vacuum-induction melting, although rupture ductility is improved significantly; and 6) the improved homogeneity of segregation-sensitive hardenable stainless and iron-base superalloys that results from consumable-electrode vacuum-arc remelting leads to improved tensile strength, ductility, fatigue strength, and stress-rupture properties.

Order PB 151085 from the *Office of Technical Services, Dept. of Defense, Washington 25, D. C.*

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Rare Earth Research Will Be Discussed At October Meeting

A four-day seminar on Rare Earth Research is planned at Arrowhead Center, the University of California, Oct. 23-26. The seminar is being sponsored jointly by the University of California, the US Department of Defense, and Research Chemicals div. of Nuclear Corp. of America.

The program is planned as a series of five sessions, each consisting of a brief summary by an author, followed by a general discussion period. The areas of solution chemistry, ceramic systems, metals, alloys, and intermetallics are to be discussed.

Arrowhead Center is located in the San Bernardino Mountains, on the shore of Lake Arrowhead, approximately 85 mi from Los Angeles. The Center has lodge facilities for 100 attendees; so, attendance is restricted to those actively interested in research with the rare earth elements. Expenses are rated at $12.50 per day (which includes room and three meals). A fee of $25 is charged for registration.

Requests for reservations should be directed to E. V. Kleber, Research Chemicals, 170 West Providencia, Burbank, Calif., or Harry Tallman, University of California, Los Angeles 24, Calif.

La Revue de Metallurgie was created in 1904 by Henry Le Chatelier. In June 1959 it changed format, now appearing in two separate monthly parts.

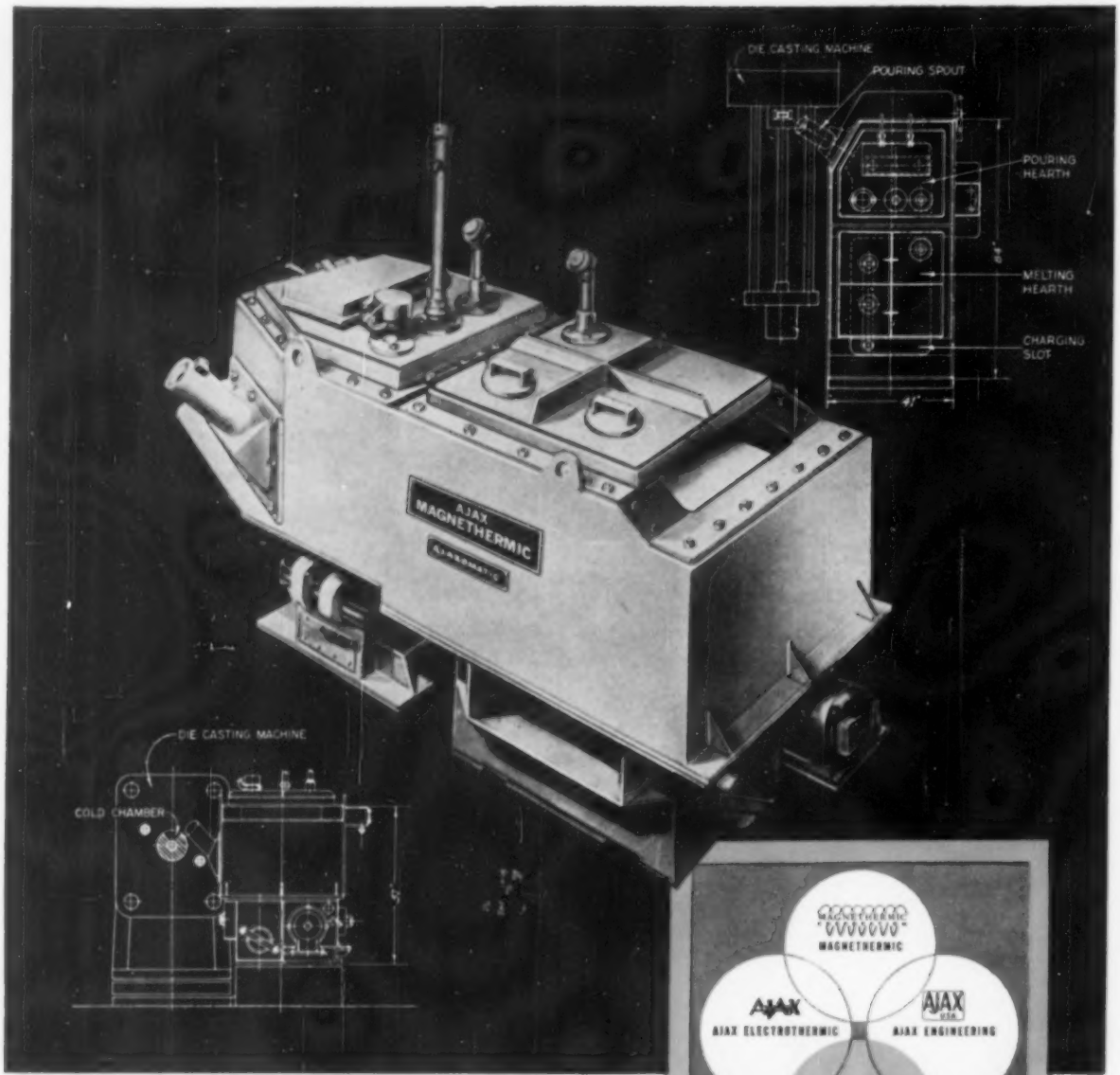
One part, which preserves the title **Revue de Metallurgie,** is devoted particularly to applied metallurgy, especially the manufacture and working of ferrous and nonferrous metals.

It also includes economic and statistical reports, as well as news of activities of the Societe Francaise de Metallurgie and related institutions.

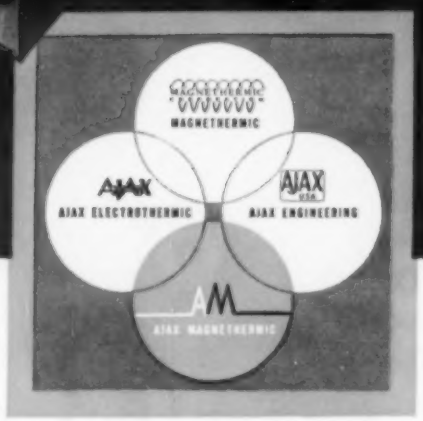
The second part of the magazine maintains the scientific character of the former publication and, as in the past, includes separate sections devoted to original papers and to abstracts. The title of this part is **Memoires Scientifiques de La Revue de Metallurgie.**

Subscription rates for 1960

Revue de Metallurgie	$16.50
Memoires Scientifiques de la Revue de Metallurgie	$35
Both publications jointly	$47



Duplex Ajaxomatic for melting and holding metal for automatic casting operations.



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METAL WORKING WITH EXPLOSIVES

Explosive forming is a dynamic new method of metal working. Many operations such as forming, sizing, cutting, compacting, and welding can now be performed by the energy released from detonating or deflagrating an explosive. The author presents a summary of activities to date and horizons opening for the future.

by John Pearson

During the last several years considerable interest has been generated in the use of explosives as a high energy rate source in the production of metal parts. While the explosive working of metals is still an area which is relatively new to the metals industry, it already includes such operations as forming, shaping, sizing, forging, extruding, welding, and compacting. In fact, it includes any operation in which the energy released from either a detonating or deflagrating explosive is utilized to perform work on a metal specimen in bulk, sheet, or powder form. Several of these basic types of operation are currently in use on a production basis, while the others are still in the applied research area of investigation.

The use of explosives to do constructive work with metals was initiated in the 19th century. About 1888, for example, Charles Munroe[1] used explosive charges to engrave names and various designs in iron plates by techniques similar to some of those used at the present time. In contrast to Munroe, who was limited in his choice of explosives and used pressed gun cotton for the energy source, the present day metal worker has a large range of explosives from which to choose. Following the work of Munroe, a number of other investigators performed operations which would currently be classed as explosive working. These earlier experiments attracted little attention, however, since there was no need for such operations at that time.

Explosive working has attracted current interest due to a number of reasons. Among these are 1) the need for performing press operations on large parts where the required force is greatly in excess of present press capacity, 2) the need for new techniques in working some of the so-called *space age* metals as well as for producing *space age* shapes, 3) possible savings in terms of equipment and labor for the production of certain types of short run items, and 4) a psychological factor which is very strong in this Country at the present time—the desire to seek and to participate in the development of new ideas.

JOHN PEARSON is head, Detonation Physics group, Michelson Laboratory, US Naval Ordnance Test Station, China Lake, Calif. This paper was presented at the 1960 Pacific Northwest Metals and Minerals Conference.

Energy sources

Many types of explosives, which can be used in metal working operations, are available. They come under a large number of different commercial names and military designations, and possess a wide range of properties. One of the problems facing the metal worker is to match the properties of a specific explosive to a particular type of operation in order to obtain the best results.

The major breakdown of explosive types, which is most pertinent to metal working, is into high explosives and low explosives. Both types are used extensively in explosive working operations. A high explosive can be typified by TNT, PETN, Composition B, or dynamite. Common examples of low explosives are smokeless powder and black powder.

Table I gives a comparison for some of the important parameters for both high and low explosives. A high explosive detonates producing extremely high pressures, in the order of 2,000,000 to 4,000,000 psi. The conversion time, i.e. the time required to convert a working amount of high explosive into high-pressure gaseous products, is measured in microseconds. A low explosive burns or deflagrates, producing much lower pressures, in the order of about 40,000 psi. The conversion time for a low explosive is measured in milliseconds or greater.

Upon initiation of detonation or deflagration, an explosive charge does not instantaneously release all of its energy to the surrounding medium. Rather, there is a finite time during which the explosive is converted into the high-pressure, high-density, gaseous products of detonation. The velocity with which this conversion front moves through the explosive is known as the detonation velocity for high explosives, and the deflagration velocity for low explosives. For most high explosives this value is in the range of 6,000 to about 28,000 ft per sec. Table II indicates representative values for the detonation properties of several of the higher-energy type of high explosives. For low explosives the deflagration velocity is usually measured in terms of inches per second or several feet per second. A thorough treatment of the physics of explosives is given in Ref. 4.

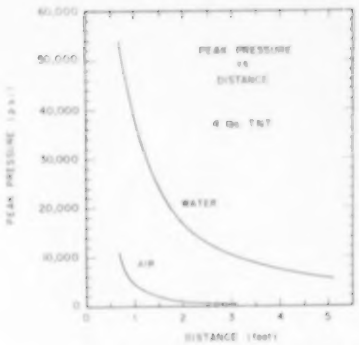


Fig. 1—Left, peak pressure as a function of stand-off distance. (2 media)

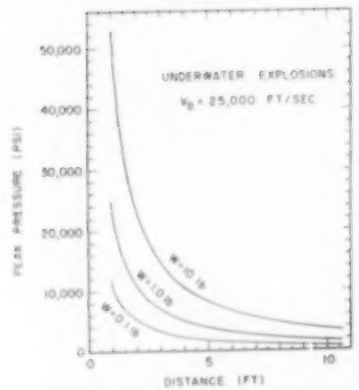


Fig. 2—Left, peak pressure at the workpiece as a function of charge size and stand-off distance.

Table I. Characteristics of High And Low Explosives

Property	High Explosives	Low Explosives
Method of initiation	Primary high explosives—ignition, spark, flame, or impact. Secondary high explosives—detonator, or detonator and booster combination.	Ignition
Conversion time	Microseconds	Milliseconds
Conversion rate	6,000 to 28,000 ft per sec.	A few inches to a few feet per second.
Pressures	Up to about 4,000,000 psi.	Up to about 40,000 psi.

Table II. Properties of High Explosives (After Ref. 2 and 3)

Explosive	Detonation Temp (°C)	Detonation Velocity (ft/sec)	Detonation Pressure (psi)
RDX	5450	27,400	3,750,000
PETN	5400	27,200	3,300,000
TETRYL	4400	25,700	2,900,000
PICRIC ACID	3900	24,100	2,650,000
TNT	3900	22,800	2,250,000

Table III. Velocities of Elastic Waves in Materials

Material	C_L (ft/sec)	C_P (ft/sec)	C_R (ft/sec)	C_T (ft/sec)
Aluminum	20,900	17,800	16,800	10,200
Beryllium	42,300	42,200	42,200	29,100
Brass	14,100	12,500	11,500	6,700
Copper	14,900	12,800	11,700	7,400
Lead	7,100	4,300	4,000	2,300
Magnesium	21,100	17,800	15,700	10,100
Molybdenum	20,900	19,850	18,000	11,200
Nickel	18,800	16,800	16,300	9,800
Steel	19,500	17,800	16,400	10,500
Tin	9,500	8,300	8,200	4,800
Titanium	19,700	16,900	15,900	9,700
Zinc	12,300	11,900	11,600	7,500

In any metal working operation, the impulse delivered to the workpiece by the energy source is of prime importance. As shown by Table I, large differences exist in the values for pressure and time between high and low explosives. Due to the extremely high pressures produced by high explosives, they are normally fired as bare charges in open systems, and the loading curve is represented by a high-intensity pulse of relatively short duration. Low explosives produce much lower pressures which can be sustained for longer periods of time. As a result, low explosives are normally used in enclosed systems where the containment helps to increase the time for which the pressure acts—hence, increasing the impulse delivered to the workpiece.

Forces and pressures

Most of the explosive metal working operations in use or under consideration can be divided into two main groups, depending on the position of the explosive charge relative to the workpiece. The first group consists of those operations in which the charge is placed in intimate contact with the workpiece and are referred to as *contact* operations. The second group includes those operations in which the charge is located some distance from the workpiece, and the energy is transmitted through an intervening medium. These are classed as *stand-off* operations. Large differences in the magnitude of the pressures developed at the workpiece, and variations in the resulting material behavior, will occur between these two classes of operations. While most production parts are currently made by stand-off operations, the interest in contact operations is increasing rapidly.

When a high explosive charge is placed in intimate contact with a steel body and detonated, pressures as great as 4,000,000 psi may be developed at the metal-explosive interface*. Pressures of this magnitude are developed wherever the explosive is in contact with the metal. This means that the basic restriction in the magnitude of the force which can be developed is related to the surface area of the metal that is exposed to direct explosive contact. For large contact areas, the magnitudes of the forces involved become so large as to be essentially meaningless, since it is almost impossible to grasp the physical concept of the action. Their meaning, in terms of available work on the body, is further complicated by the fact that load duration is measured in microseconds.

In the great majority of explosive working operations currently in use, pressures in the order of several million psi are far in excess of what can be utilized. Lower pressures are obtained by placing the explosive charge some distance from the workpiece and using an energy transmittal medium such as air or water between the charge and workpiece. Parameters which are important in controlling pressure magnitude at the workpiece include: 1) the type of explosive, 2) the amount of explosive, 3) charge configuration, 4) stand-off distance, 5) the type of energy transmittal medium, and 6) the possible effects of pressure confinement. By varying these parameters in stand-off operations, working pressures of several thousand psi to several hundred thousand psi may be obtained.

* It should be realized that the value of 4,000,000 psi is only approximate. Contact pressures will vary over a large range depending upon the type of explosive used and the properties of the metal with which the explosive is in contact.

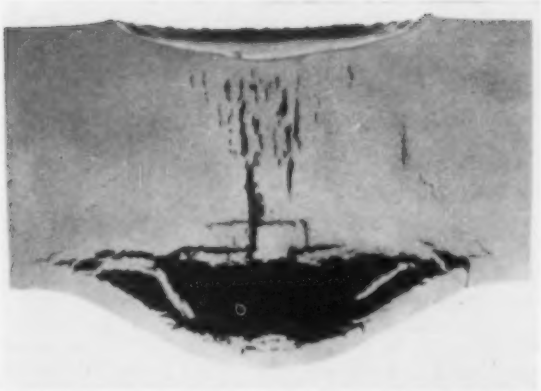


Fig. 3—A cross-sectional view of a steel plate—the bottom surface fracture is due to a scabbing or spalling process. A stress wave induced fracture.

Fig. 4—A small-diameter explosive charge was used to remove a conical hub from one end of the thick-walled brass cylinder.

Fig. 1 indicates how peak pressure at the workpiece can be varied as a function of stand-off distance for two transmittal media: air and water. Pressure curves of this type for air can be obtained from equations given by Stoner and Bleakney[3], Cook[4], and others. For a given amount of high explosive, the peak pressure at a given distance from the energy source is many times greater in water than in air. Several other important differences between pressure effects in air and water exist which are not indicated by the curves of Fig. 1. When a high explosive charge is detonated in air, the workpiece is usually subjected to a single loading pulse with a duration measured in microseconds. When a charge is detonated underwater, an oscillatory bubble effect may be produced, if the water container is strong and relatively large compared to the diameter of the bubble. Thus, the underwater workpiece may be subjected to a series of loading pulses of rapidly decreasing pressure. Even for a single underwater pulse, the duration of load is usually measured in milliseconds.

Fig. 2 shows the variation of peak pressure at the workpiece as a function of charge size and stand-off distance. In this case, the transmittal medium is water, and the calculations are based on a high energy explosive such as Composition c-3 with a detonation velocity of about 25,000 ft per sec. For calculations such as these, the following types of equations given by Cole[4] or Roth[7] can be used:

$$P = K\left(\frac{W^{1/3}}{R}\right)^{\alpha} \qquad [1]$$

$$P = 155\left(\frac{W^{1/3}}{R^{1.15}}\right)\sqrt{V_D} \qquad [2]$$

where P is the peak pressure in psi, W is the weight of explosive in pounds, R is the distance from the explosive in ft, V_D is the detonation velocity of the explosive in ft per sec, K is a constant which varies with the explosive, and α is a constant which is generally taken as 1.15.

The total impulse imparted by an explosive load to a workpiece is represented by an integral effect of pressure and time,

$$I = \int p\,dt. \qquad [3]$$

Hence, for a given impulse the shape of the pressure-time distribution can take countless forms. In many operations, this shape appears to be of considerable importance in obtaining satisfactory results, and different magnitudes of impulse and different p-t distributions may be desired. However, since both parameters (pressure and time) are amenable to some control over relatively large ranges, the use of explosive charges makes available a tremendous work range within which metal working operations can be performed.

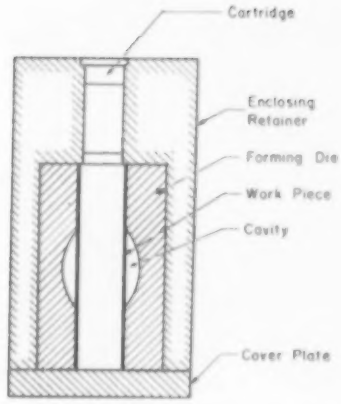
Stress waves

General Effects

In cases where the explosive charge is detonated in contact with the workpiece, much of the energy of the detonating explosive is coupled into the metal and appears in the form of a high intensity stress pulse. These stress waves can be either destructive or beneficial, depending upon how their energy is utilized in the system. The outstanding behavior factor associated with stress waves is the formation of tensile fractures through the reflection and interaction of stress wave fronts. The metal specimens in Fig. 3 and 4 show types of stress-wave induced fractures which may be either desirable or undesirable, depending upon the nature of the operation.

Fig. 3 shows a representative type of fracture which frequently accompanies contact operations and which is normally undesirable. The figure shows a cross-sectional view of a steel plate, which had been loaded on the upper surface with a small explosive charge. The fractures on the bottom surface of the plate are the result of a process known as *scabbing* or *spalling*. Such fractures are produced when a high intensity compression wave strikes a free surface and is reflected back in tension. The superposition of the reflected tensile front on the incident compressive tail produces a localized region of high tensile stress in the metal. If this tensile stress is greater than the critical dynamic fracture strength of the metal, fractures of the type shown in Fig. 3 will result. The dynamics of formation for such fractures is well known[8, 9], and fractures of this type normally can be avoided by proper design of the metal-explosive system.

Fig. 4 shows a large, thick-walled brass cylinder where a conical hub was removed from one end by means of a small-diameter explosive charge detonated along the axis of the cylinder.[10, 11] In this case, the conical fracture surface was produced by the controlled interaction of tensile stress wave fronts. This specimen is representative of a large number



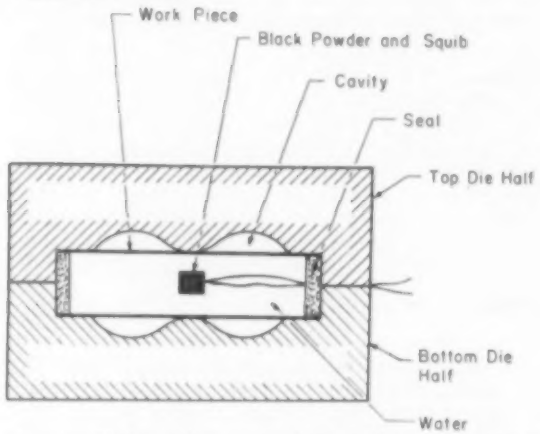


Fig. 5—Right, and Fig. 6—below, illustrate two bulge forming operations.

of controlled cutting operations which are possible through the use of explosively-induced stress waves. The dynamics of formation of fractures of the above type is extensively discussed in Ref. 8.

Physical Properties

The velocity of a stress wave depends both upon the properties of the material and the configuration of the body through which it is propagating. The velocities, as well as the general physical behavior of stress waves, are quite well known.[8, 9, 12, 13] Velocity values for stress waves in several representative metals are listed in Table III. The first three columns give the velocities of longitudinal waves for three basic, body configurations: bulk, plate, and rod, respectively. The last column gives values for shear or transverse waves. Most of these values were experimentally obtained and come from numerous sources.

Several of the most useful equations for expressing stress wave velocities are as follows:

The velocity of propagation of longitudinal waves (tension or compression) in a medium of infinite extent is given by the equation

$$C_L = \left[\frac{3K(1-\nu)}{\rho(1+\nu)} \right]^{1/2} \quad [4]$$

Since this represents the velocity in bulk material, it is the most useful value when working with real objects.

The respective velocities for longitudinal waves in thin plates and rods of small diameter are

$$C_P = \left[\frac{E}{\rho(1-\nu^2)} \right]^{1/2} \quad [5]$$

and

$$C_R = \left(\frac{E}{\rho} \right)^{1/2} \quad [6]$$

The latter two equations probably will find only limited use in metal working operations due to restrictions imposed by geometry and dimensions.

The velocity of propagation of transverse or shear waves in a body of infinite extent is given by the expression

$$C_T = \left(\frac{G}{\rho} \right)^{1/2} \quad [7]$$

The role of transverse waves in various working operations is not yet very well understood.

In the above equations K is the bulk modulus of elasticity, E is Young's modulus, G is the shear modulus, ν is Poisson's ratio, and ρ is the density of the material through which the wave is traveling.

Once a stress wave has been explosively induced in the workpiece, the wave will appear as a sharp-fronted pulse of high intensity. As the wave front passes a given point in the body, elements in the body will be displaced with a velocity which is termed the particle velocity.

The particle velocity V_L of a point within a longitudinal disturbance is given by the expression

$$V_L = \frac{\sigma}{\rho C_L} \quad [8]$$

where σ is the stress at the point. For a compression wave, particle motion is in the same direction as that of the wave. For tension wave, the direction motion is opposite to that of movement of the wave.

The particle velocity of a point within a plane transverse wave is given by the expression

$$V_T = \frac{\tau}{\rho C_T} \quad [9]$$

where τ is the shear stress at that point within the transverse wave. Particle motion within a transverse wave is normal to the direction of wave propagation.

Another expression which is of considerable importance in contact operations is relative particle velocity. This value represents the difference in velocity which exists between two adjacent body elements under dynamic conditions. If this velocity difference is known, then Equation 8 can be used to put this value in terms of a stress differential. However, the dynamic behavior of most explosively-loaded, metal bodies can best be understood in terms of particle velocities and relative particle velocities than in terms of stresses and stress differentials.

Metal working operations

General

A wide variety of explosive-working operations are currently being used, or are being studied for possible future application in the metals industry. In general, those operations which are in the most advanced stage of development are the ones which most approximate conventional metal working. These are mainly stand-off operations and include the forming, sizing, and flanging of sheet-metal stock and plate. Those which are in the least advanced stage of development are the ones most dissimilar to conventional operations and frequently involve different concepts of metal behavior. Most of these are contact operations and include controlled work hardening, forging, extruding, welding, controlled cutting, and the compacting and forming

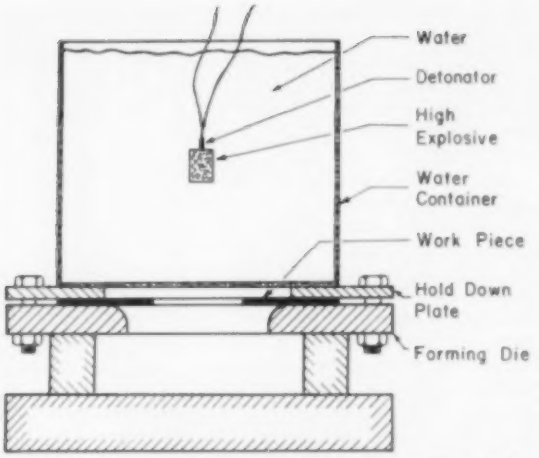


Fig. 7—Shows a general method for forming large open parts in a water pit.

of powders. In the following sections several representative explosive metal working operations of both the stand-off and contact variety are discussed.

Forming, Sizing, Flanging

The most extensively used explosive metal working operations at the present time are the forming, sizing, and flanging of sheet metal stock. The equipment involved, and the techniques applied to these three areas are frequently quite similar. These operations are normally considered as stand-off operations with air, water, oil, and occasionally other materials used as the energy transmittal media. Water is used predominantly, and both low and high explosives are used as energy sources. Low explosives predominate in closed systems, and high explosives in open systems.

One common type of forming operation, which has many modifications, involves the use of a two-piece die with the workpiece enclosed. Fig. 5 shows one method used for bulge forming tube stock. The energy source is a powder cartridge inserted and fired at the top of the die enclosure; the transmitting medium is air. A thin layer of a plastic material, or a layer of thick grease, is frequently used to protect the inside surface of the part from any possible scouring action of the powder products. Fig. 6 shows a two-piece die operation used for bulge forming with a water medium. The black powder charge and squib encased in a suitable container are placed in the center of the workpiece which is then filled with water and sealed at both ends. For a relatively thin-walled cylinder a single charge as shown is effective but for larger, heavier-walled parts it is often desirable to use multiple charges, each one located at the center of an area of heavy deformation.

Fig. 7 shows a general method for forming large open parts in a water pit. For such operations a high-explosive charge is most commonly used. The charge size, stand-off distance, and water head are parameters which are determined by the size, thickness, and material properties of the workpiece as well as by the depth of draw. The workpiece is placed in the die assembly above water; the assembly is then lowered to the bottom of the tank for firing. In such operations, a hold-down ring is normally required to prevent wrinkling of the part.

For underwater operations some type of seal is normally used between workpiece and die. Some

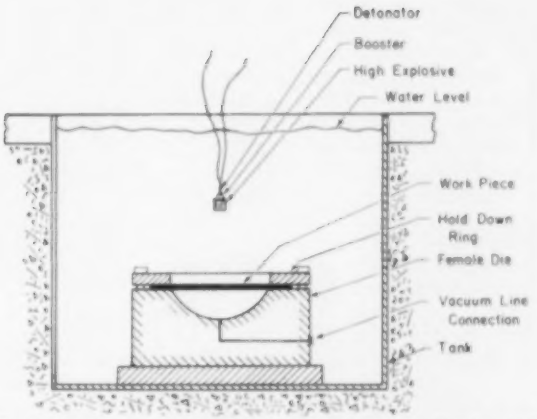


Fig. 8.—Depicts a method which is applicable to both flanging and free forming with explosives.

producers use rubber *O* rings in the top surface of the die. Others use a light layer of grease or other material between the die surface and the workpiece. Still others report that no supplementary seals are used. Apparently, in the latter case pressure applied through the hold-down plate serves this purpose. The need for a vacuum in the die cavity is another variable in question. Most operators report using a vacuum; a few do not. Whenever a vacuum system is not employed, special venting techniques are used.

Operations of the type shown in Fig. 7 are frequently performed without the use of a water pit. The explosive charge is then placed in an expendable, water-filled cardboard container which is positioned on top of the hold-down ring or workpiece. This method is quite adequate for the drawing of small parts; however, a water pit is almost a necessity for the production of large parts.

Explosive sizing operations may be employed in several ways. The workpiece is first preformed either by explosive or conventional methods, and then is sized to shape by explosive techniques similar to those described previously. Explosive sizing has also proven to be a useful tool for saving rejects when large parts produced by conventional methods fall outside of tolerance limitations. Another unique application is to take large cylindrical tubing which normally comes with relatively large variations in dimensions, and use explosive sizing to bring it within close tolerances for special uses.[14] One of the main advantages of explosive sizing is the ability to hold close tolerances in the workpiece.

Some of the simpler flanging operations are performed in the same manner as for free forming with explosives. Fig. 8 shows a method which is basic to both types of operations. In this case a high explosive is placed in a water-filled cardboard container sitting on the hold-down plate. For a free-forming operation the workpiece would be a solid sheet or plate. Free-forming operations such as this are occasionally used to preform the workpiece, with the final forming operation similar to that shown in Fig. 7. A flanging operation can be performed if the workpiece in Fig. 8 has the center suitably removed; however, more complicated shapes may require a more complex system than that shown.

The parts shown in Figs. 9, 10, 11, and 12 are representative of types and sizes currently being produced by the above operations. It is in this area

of parts production that the greatest number of pieces are now being manufactured. Operations of the above types have been effectively used to produce cylindrical parts ranging from a fraction of an inch to several feet in diameter. Corresponding part lengths range from several inches to several feet, with material thicknesses of from a few thousandths of an inch to about one inch being used.

The pressures encountered in most stand-off operations of the type described in this section range from about 20,000 psi to about 200,000 psi. Load duration for these operations is mostly in the millisecond range or longer. The displacement velocity of workpiece elements is large compared to conventional forming, but it is relatively slow when compared to ordnance velocities. For example, in the free forming of a sheet metal part, the maximum displacement velocity for any part of the piece is normally in the range of from 100 to 800 ft per sec. When the dynamic aspects of such a loading are considered in terms of the geometry of the system, the relative particle velocity between adjacent elements of the workpiece becomes a matter of only a few feet per second. This is normally well below the critical impact velocity of the metal.

Compacting and Forming of Powders

The use of explosives for the working of powdered metals falls into several categories. Among these are operations where: 1) a high density packing is required without changing the physical properties of the individual particles, and 2) it is desired to compress the powders into actual parts through the bonding together of the particles.

One of the earlier forms of powder compaction consisted of placing the powder in a thin cloth or plastic container and subjecting it to the air blast from an explosive charge. A sequence of shots, or multiple simultaneous shots were sometimes used. In these operations the powder could be given a high-density packing without metallurgically bonding the particles together. Another general method for getting a high-density powder compact involved the concept of a closed system and the use of low explosives. Pre-tamping and the use of vacuum systems helped in the compacting of powders.

To obtain the pressures required to form the powders into actual parts, contact charges can be used with the energy from the explosive being transmitted to the powder through some type of metal buffer. Systems of this type have been used mainly as research tools for studying the behavior of material properties. However, the concept for these research tools appears applicable to part production through modification of the system design.

Two types of explosively-activated presses which have been used at the US Naval Ordnance Test Station utilize the principles of single and double pistons.[15,16] Fig. 13 shows a cross-sectional sketch of a simple single piston press where the powder is placed in the cavity of the lower platen. A high-explosive charge placed at the center of the upper platen forcibly drives the piston into the cavity. Higher pressures can be obtained with double piston presses. Fig. 14 shows a typical press of this type where the charges are detonated simultaneously by means of two detonators and a high-voltage system. Fig. 15 is a representative pressure-time curve for a small double piston press using ½ in. diam pistons and Composition-B explosive charges 1 in. diam x 1 in. long.[17] Fig. 16 shows typical parts which can be produced by explosively-activated presses, and the different sets of forming pistons employed.

Another application of explosive pressing consists of the bonding of layers of dissimilar materials. Fig. 17 is a photomicrograph of the interface formed by compressing a layer of iron filings upon a layer of titanium filings.

To date, most explosive press work has been of an exploratory nature and little quantitative data are yet available for publication. Measurements on specimens formed in double piston presses have shown that the final density and the strength of the specimen are closely related to the ductility of the material pressed. In general, powders of a ductile material will undergo considerable plastic flow and have a tendency to form a strong bond. Extremely brittle materials normally undergo a decrease in particle size and demonstrate good compacting properties, but produce little bonding strength. In many operations the desired properties of a brittle material may be utilized by press-bonding such a powder into a more ductile matrix. Also, the use of sintering techniques in explosive press forming has not yet been adequately studied, but is one of many areas which seem to offer unusual opportunities.

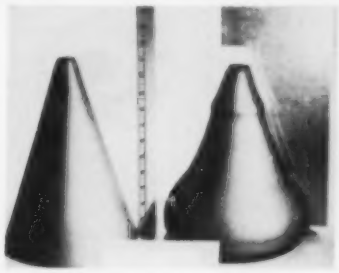
Explosive Welding

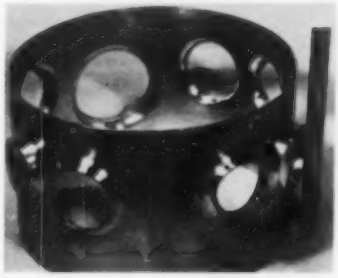
One of the most recent applications of explosives is in the welding of metal parts. Only limited preliminary studies have been reported in this area.[15,18]

It appears that two basic explosive welding techniques are currently being considered. In one method the parts are placed in direct contact and are subjected to normal loads applied by an underwater stand-off operation. The second method uses contact charges, asymmetrical loads, and an angularity between parts. Little information is yet available with which to compare the strength characteristics, etc., of these two techniques. But both methods have demonstrated promise in the joining of dissimilar metals, and it appears that the two methods will have specific but different areas of application. Only the contact explosive method is discussed here.

Consider the four systems shown in Fig. 18. All have the tendency to produce metallic jets. In the ordnance-type shaped charge the metal liner collapses and is projected forward as a jet of metallic fragments. When a high-velocity projectile, such as a sphere, impinges on a thin stationary target, there is a tendency for the metal at the interacting surfaces of the sphere and the target to produce a jet. This unusual effect between target and projectile was described by Allen and associates[19] and at a later date by Abrahamson.[20] Fig. 19 shows the type of wave effect produced on the surface of a steel projectile by impacting a thin lead target.[20] This effect is termed *surface jetting*.

When two plates or other parts are projected together at a high velocity with some included angle of collapse, surface jetting occurs from the interacting surfaces. If the impacting velocities are too great, relative to the properties of the metal, the material at the interface will be displaced as a jet and bonding between the parts will not occur. If the velocities are too low, no interaction will occur at the interface. Between the two extremes is a velocity region within which severe plastic deformation occurs at the interacting surfaces of the two parts, and surface jetting similar to Fig. 19 results. The interaction between the jet elements of the two surfaces, followed by a rapid release of load, leaves the





Figs. 9, 10, 11 and 12—Show representative types and sizes currently produced by forming, sizing, and flanging operations.

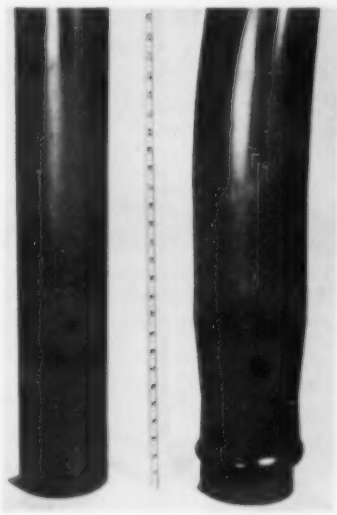
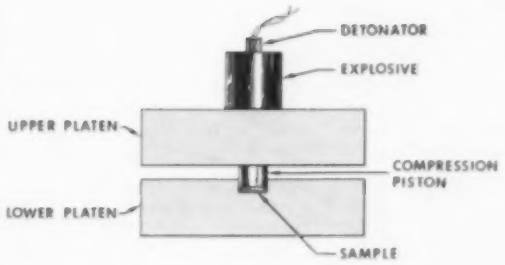
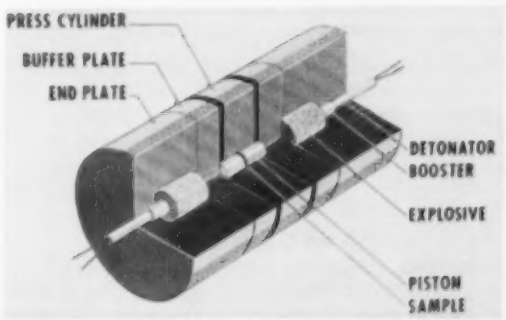
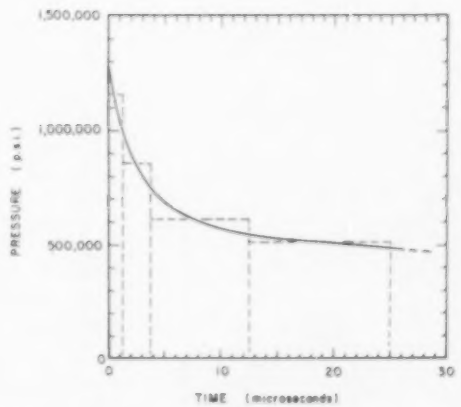


Fig. 13—Left, shows a cross-sectional sketch of a simple single piston press where powder is placed in the cavity of the lower platen.

Fig. 14—Below, shows a typical double piston press where the charges are detonated simultaneously.







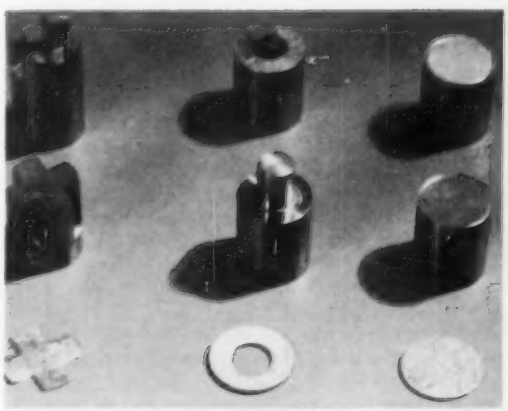


Fig .15—Above, shows a representative pressure-time for a small double piston press. Fig. 16—Right, shows the various parts which can be produced using presses and forming pistons now available.

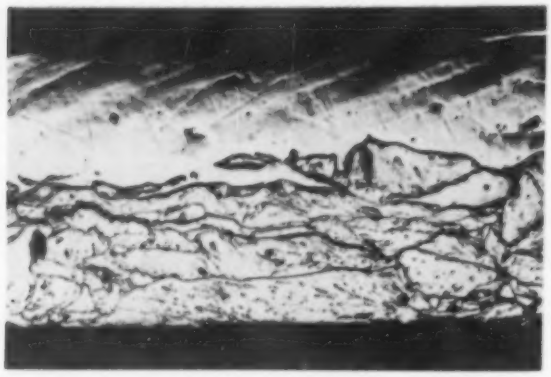


Fig. 17—Above, a photomicrograph of the interface formed by compressing a layer of iron filing upon a layer of titanium filing.

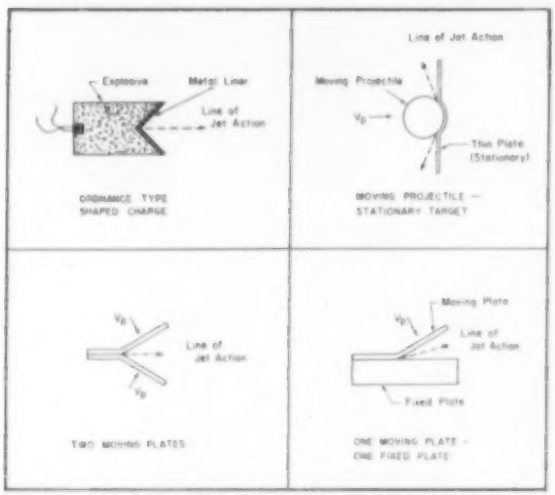


Fig. 18—Four systems which tend to produce metallic jets.

two parts permanently bonded together. Fig. 20 shows the cross section of the interface between two low carbon steel parts which have been joined together by surface jetting. Limited tests on explosively welded plates indicate that the strength of a good weld of this type is comparable to the strength of the parent metal.

Other Operations

In addition to the operations previously mentioned, many other explosives applications of both the stand-off and contact charge variety are being used. Embossing, beading, dimpling, and joggling operations are normally performed with stand-off charges in a manner similar to those described for the general forming of sheet metal parts. Engraving, extruding, and punching are types of contact operations where buffer parts in the form of engravement sheets, pistons, etc., are placed between charge and workpiece. In most controlled cutting operations the charge is placed in contact with the workpiece, and the metal is parted through the interaction of stress waves. In some instances cutting is done by means of specially designed shaped charges with metal liners. Most of these operations are fully described in the literature.[22-26]

Controlled hardening of metal parts is another recent and novel development. Contact charges are used to increase the hardness of austenitic manganese and other steels.[27]. The application of this process at present is used mainly for railroad, construction, and mining equipment. Special types of sheet explosive have been developed for this application. Other data on the explosive hardening of mild steel is also available in the literature.[28-29]

Material behavior

The behavior pattern of metals in explosive working operations is still a matter for considerable debate. Much confusion stems from the fact that a wide range of metals are being considered, and for many diverse operations. It is in this general area that the greatest amount of applied research needs to be accomplished.

Much of the current information regarding the general behavior of metals under high-intensity loads comes from the earlier work of such investigators as Clark and Wood, and Bridgman. The studies of Clark and Wood and others on the behavior of tensile specimens subjected to dynamic loads,[30] demonstrated that an increase in the rate of deformation will raise the yield strength, as well as the entire stress level of the flow curve, and the ultimate strength of the material. Dynamic tensile tests are not as clear with regard to elongation characteristics. With increasing impact velocity some metals show essentially no increase in elongation, others have a scattered behavior, while still others show a definite increase or decrease in elongation. A similar spread in behavior patterns has been reported for a wide range of metals when subjected to stand-off operations. Other favorable predictions for certain types of explosive working operations are based on the static high-pressure work of the type conducted by Bridgman.[31] The general outstanding effect associated with this work on such metals as low carbon steels is the tremendous increase of plastic deformation which the metal will tolerate without fracture.[32] Similar behavior patterns have been reported for a few contact explosive operations where unique load geometries were applied.

All such data is of considerable value, provided that it is interpreted correctly in terms of explosive working parameters. In attempting to extrapolate such data to any explosive working operation, the investigator must be careful to account for the marked differences in loading patterns which may exist between these two areas. For example, unusual behavior patterns exhibited under high hydrostatic pressure may, or may not, exist in an explosive working operation, depending on the nature and geometry of the load.

It is helpful to divide material behavior in explosive working operations into separate areas for consideration. One useful breakdown is shown in Fig 21. The basic breakdown is into the areas of stand-off and contact operations which are then arranged in order of increasing pressure and strain rate. Recent studies indicate that for most standoff operations (the left end of the scale), the problems

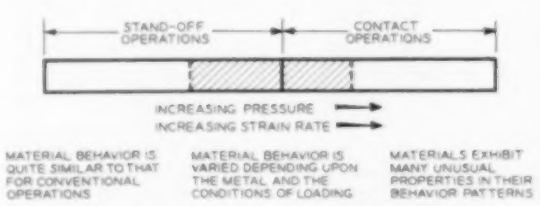

STAND-OFF OPERATIONS
CONTACT OPERATIONS
INCREASING PRESSURE
INCREASING STRAIN RATE
MATERIAL BEHAVIOR IS QUITE SIMILAR TO THAT FOR CONVENTIONAL OPERATIONS
MATERIAL BEHAVIOR IS VARIED DEPENDING UPON THE METAL AND THE CONDITIONS OF LOADING
MATERIALS EXHIBIT MANY UNUSUAL PROPERTIES IN THEIR BEHAVIOR PATTERNS

Fig. 21—A helpful division of materials behavior in explosive working operations.

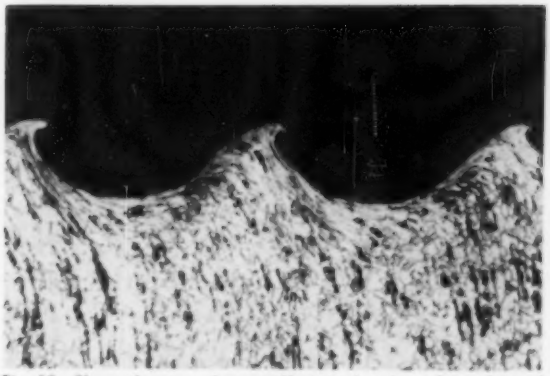


Fig. 19—Shows the type of wave effect produced on the surface of a steel projectile by impacting a lead target.

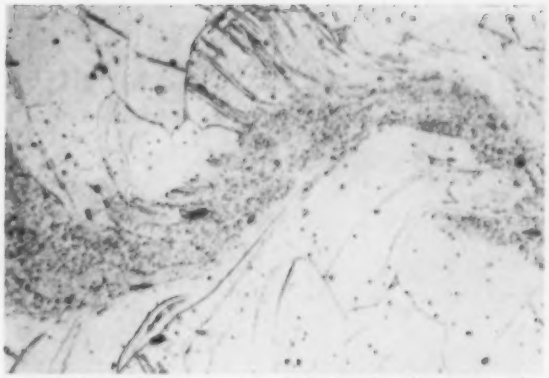


Fig. 20—Shows the cross section of the interface between two low carbon steel parts which have been joined by surface jetting.

encountered in metal behavior are similar to those of conventional working. For most metals in this area of operation the degree of work hardening, elongation, strength characteristics after working, etc., are not much different from what would be encountered from similarly working the part by conventional methods. At the right end of the scale where most contact operations are listed, materials tend to exhibit stress-wave induced fractures, extensive plastic deformation, severe work hardening, and extensive flow and elongation. Many operations are designed around these behavior patterns and use them to advantage. In the region between these two extremes no single behavior pattern appears to hold. Material behavior is quite varied, depending on the metal and the conditions of loading.

Summary

The use of explosives in metal working has been satisfactorily demonstrated during the last several years. What have not yet been established, however, are the limits of application in various operations coupled with the workability of a wide range of metals. It is a young field with much of the work being accomplished on an empirical basis. To help it grow a greater interchange of information is needed, as well as a much greater emphasis on applied research.

As with most techniques that are new, emphasis has been placed on reproducing types of operations that are understood in the conventional sense. Many of these operations, especially those used in the forming of large sections of high-strength, hard-to-form materials, and in the production of low-volume, unusual shape items, have been outstanding. However, explosive working is not only a new way of doing an old job, but one which also offers new and unique operations of its own. In fact, the future promise for this field seems to lie in the extension of explosive working to operations now beyond the reach of conventional equipment, rather than in duplicating the results of conventional methods.

Explosive working is not a cure-all for all of the problems which exist in the conventional working of metals, although it does offer help in many cases. In many operations it is not a replacement for the older method, merely an adjunct to it which allows for greater scope of operation. In other instances it promises revolutionary new techniques all of its own. It is safe to say that if a job can be satisfactorily and economically performed by older, more conventional methods, then explosive working is not needed. However, if conventional methods are not now doing a satisfactory job, then explosive working should be seriously considered; in many cases it will offer a satisfactory solution.

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HIGH ENERGY RATE...

its application to metal forming

Promising the engineer unfettered design freedom, explosive, hydroelectric, and pneumatic-mechanical forming—the most important high energy rate processes for the deformation of materials—are explored in detail. Explanation is also given to a new concept, closed-cavity extrusion molding.

by E. W. Feddersen

Three major methods of applying high energy rates to the deformation of materials are explosive, hydroelectric, and pneumatic-mechanical forming.

Explosive forming is the shaping of materials by the exertion of sudden pressures from the explosion of a chemical charge. The shock wave and subsequent hydraulic action cause the material to take the shape of its die. Normally, a fluid medium, such as water, is used to transmit the force because of the intense heat generated and the more uniform pressure is experienced from hydraulic action. A third and a less important reason for using water is the natural muffling effect that it has on the sound of the explosion.

Although possessing certain advantages over mechanical presses, explosive forming cannot replace mechanical means of forming high-volume items. The real advantage lies in its ability to form and size large, complex sections at a fraction of the normal tool costs and to eliminate extremely large machinery.

The hydro-electric method has been characterized as *underwater lightning*. Again, a sudden release of energy—in this case electrical energy stored in capacitors—is used to deform materials. The energy is applied through a water medium, and like explosive forming, this method also has its limitations and does not appear to be a replacement for the mechanical press. But the hydro-electric process has two additional advantages over the first method: it is accurate and repeatable, and because of complete controllability, it is usable within a fully-inhabited factory.

The third method is a pneumatic-mechanical means of applying high energy rates to the deformation of materials through the sudden release of compressed gas; it is called *Dynapak*. Energy is stored in the form of compressed, dry nitrogen, which is released through a system of valves and applied to a piston. The piston, ram, and header plate are driven at a very high velocity, making contact with a bolster plate. The header plate contains the punch, while the bolster plate holds the die. This is very conventional, with the exception of the high velocities which are employed. The method opens new avenues of metal forming and can, in some instances, use simple punches with complicated dies.

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Applications of explosive forming

The field of explosive forming is the forerunner of the three methods, primarily, because it does not require mechanical or electrical devices other than those employed for handling materials and tools.

Production applications of explosive forming is most prominent in the aircraft, missile, and engine accessory fields. Ryan Aeronautical Co., Aerojet General Corp., North American Aviation Inc., Rocketdyne div. of North American Aviation Inc., Lockheed Aircraft Corp., Boeing Airplane Co., and Convair installed facilities for research and production as early as 1957. In addition, Talco Engineering Co. and E. I. Du Pont de Nemours are currently using explosive forming methods.

Imaginations ran rampant when explosive forming was first introduced to industry. At first, everyone believed that the then conventional methods would be replaced by the easier and cheaper method which used explosives. Numerous articles were written and as many speeches were made, each emphasizing that this was the panacea that the metalworking industry had been seeking. Approximately three very active years have passed since the first blast of publicity, and during this time, many among us have quiescently become wiser and assumed a practical approach in the use of this very important and versatile form of energy.

After the first shock wave subsided, Convair-Ft. Worth initiated technical studies and tests on explosives and their capacity to deform materials.

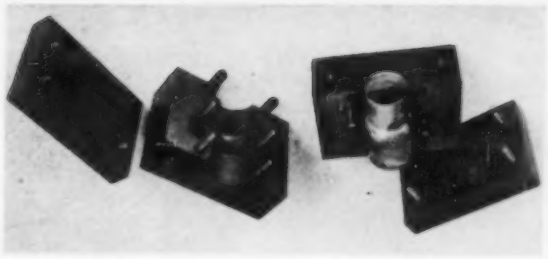


Fig. 1—Left, a ball joint—explosively formed. Fig. 2—Above, die used to form the ball joint. The explosive used was prepackaged gun powder.

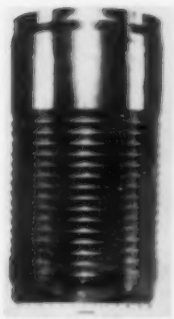
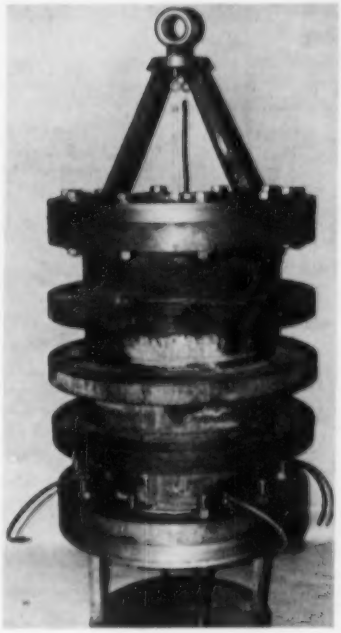


Fig. 3.—Above, shows a section of a cooling shroud for a ramjet engine. Fig. 4—Right, the die used to form the section.



These studies produced a great deal of technical data, the compilation of which has permitted Convair to establish parameters of explosive type, shape, and weight to do a given forming operation. These parameters are relatively broad, and we still rely heavily upon the engineer's practical knowledge of explosives and their characteristics. Continued use of explosives will generate more empirical data to be utilized in establishing more precise parameters.

In addition to the above data, we began a systematic classification of shapes which we consider to be practical to be formed by explosives; they include the following: preformed hollow bodies (ultimate shapes), cups (drawn from flat plates), channels (straight and curved), and panels (flat and contoured). The expansion of preformed hollow bodies and the formation of cup shapes were selected as the most economical applications of this process.

Fig. 1 shows one application of explosives to the expansion of preformed hollow bodies. The ball joint is made by precutting 301 annealed stainless steel tubing and nesting one within the other. These are then placed in the die shown in Fig. 2. A light charge of prepackaged gun powder is placed inside the tube and detonated. The result is a uniform semi-air-tight ball joint for the overboard dump system of the B-58 airplane.

Another application of explosives to preformed hollow bodies is shown in Fig. 3. This part is a cooling shroud for a ramjet engine. In this case, a type-310 stainless steel was preformed into a cylinder and welded. It was placed in the die shown in Fig. 4, and a cylindrical charge of PETN explosives, 2 in. in diam and 9 in., long was suspended within the chamber. Thus, a complicated part was formed without the need of any machine tools. Conformation of convolutions and diameters were well within customer's tolerances (± 0.010 in.).

Fig. 5 is an irregular cup shape which was selected for explosive forming because of the multiple strike and annealing operations which were required when the part was made on a drop hammer. By means of explosive forming, operations were reduced from five strikes to three and from four anneals to one. The part, a mounting for a ground cooling line on the B-58, is made from 6061-0 aluminum and is formed using two 5-g explosive charges with only one annealing required between strikes.

Fig. 5—The use of explosive forming decreases the number of steps in the production of this cup.

Fig. 6—Because of safety regulations some explosive forming installations are out of doors.

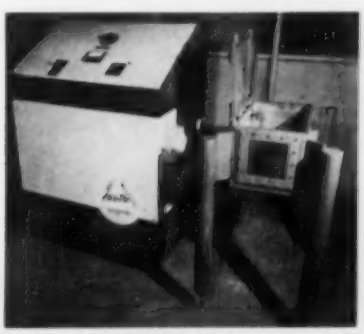


Fig. 7—A HI-VO-PAC (high energy spark) unit is shown above. The portable box on the left contains capacitors, switches and a variac power control.

The explosive forming installation, Fig. 6, is out of doors, because of safety regulations established by state and local governments and company policies. These regulations, while absolutely necessary, cause the process normally to be a fair-weather operation. With these limitations plus the fact that the blasting site is generally located some distance from the main plant facility, explosive forming is economical only when applied to selected parts. The search is continuing to find a practical method of generating controlled explosive forces of a desired magnitude, and yet safe enough to detonate in the plant.

Hydroelectric forming

Within the past two years, Chrysler Corp., Republic Aviation Corp., and Convair-General Dynamics Corp. have announced that they have found a substitute for explosives in high energy rate deformation—electricity!

Convair engineers developed a high-energy spark unit as a non-chemical high energy release method to supplement explosive forming in the study of shock waves and wave forms. Numerous experiments and tests were performed, during which it became apparent that a distinct and vast source of controlled high-energy was available. Here was a technique that could be used indoors. Here, too, was one of the few direct applications of electricity to do work.

At first, high voltages in a range from 14,000 to 22,000 v and from 30 to 60 microfarads were used. Subsequent experiments showed that a sufficiently intense shock wave could be generated in water to form small sample parts of stainless steel, copper, and aluminum. Pressures obtained were in excess of 80,000 psi—well above the yield limits of the materials used in the experiments.

As additional experiments were tried, it became essential that the power of the unit be increased. This was realized by reducing the voltage from 22,000 to 4,000 v and increasing the capacity from 60 to 1200 microfarads with the addition of an initiating wire. An added bonus derived from the reduction of voltage was the complete elimination of leakages in the form of a corona, thus making a safer operating unit.

The energy contained in the capacitor bank is equal to 14,580 joules or 10,800 ft lb. When this unit is fired, the initiating wire connecting the electrodes is transformed into a cylinder of metallic vapor, which expands against the medium, in this case water. The inertia of the medium causes a pressure build-up in the approximate range from 15,000 to 20,000 atm.

The surge peak of current is about 2 or 3 microseconds, and 40 microseconds of total time is used to decay. Variation in the wave front is accomplished by shaping the initiating wire. Sharp nosed, spherical, and plainer wave fronts have been observed and put to use for specific forming jobs.

A high-energy spark unit is shown in Fig. 7. The portable metal box on the left contains capacitors, switching circuits, a variac power control, a charging switch, and automatic or manual discharging switches. The desired voltage build-up is controlled by the variac and can be read in the voltmeter located on the control panel. After making the desired setting, the build-up and discharge is initiated merely by pressing the release button; the apparatus charges in 6 sec and then automatically discharges. The unit is sufficiently safe for operation by non-technical personnel and can be located in the proximity of other plant activities.

In the application of this controlled and repeatable electrical explosive force, the most obvious departure from explosive forming is the die design. Dies for high-energy spark forming are completely closed, because the gas volume generated by the vaporized wire is insignificant when compared to the gas bubble created by a chemical explosive. By closing the die, full advantage can be taken of the pressure wave and, in the case of tubular parts, the shock wave is reflected off the ends, thereby creating additional energy. Each die has its own peculiar set of electrodes and an initiating wire.

The closed die design, employed at Convair-Ft. Worth, permits the use of epoxies as die material. These resins have high compression strength and reasonable wear characteristics.

Fig. 8 shows the split die, blank, and diehead electrode with initiating wire attached. In the sequence of operations, the die is first closed, and the bottom plate is installed. The tube to be formed—with a rubber wafer at its base to make it water-tight—is then placed into the die cavity, and the tube is filled with water. The cover plate with electrodes and initiating wire is next installed. The leads from the power unit are connected to the electrodes, and the release button is pressed. In 6 sec the unit charges itself and fires automatically, resulting in the finished part shown in Fig. 9. Of great interest is the fact that the operations of beading, expanding, and piercing of the hole were done simultaneously.

High-energy spark, like explosive forming, obviously lends itself to expanding and sizing hollow bodies; thus, this is the most economical use to which it has been employed. This high energy rate method will take its place among the other production processes in sheet metal fabricating shops.

Pneumatic-Mechanical forming

Is there any high energy rate device suitable for producing forgings and impact and straight extrusions? The answer is to be found in a device marketed under the trade name of Dynapak. This is a pneumatic-mechanical machine which releases stored high-pressure nitrogen gas to accelerate a piston to a high velocity, thus developing a high energy rate forming process.

This pneumatic-mechanical principle offers to the metal forming industry a high energy rate device which can be used in forging, forming, extruding, compacting, shearing, and blanking. But to date, most of the effort has been in forging, extruding, and *closed-cavity extrusion molding*, a term which pertains to extruding a material through an orifice into a vented closed-cavity die. This process literally makes functional the concept of parts being *born to shape*.

Dynapak consists of a two-chambered cylinder, valve cocking assembly, piston ram assembly, bolster plate, tie rods, cushioning piston, nitrogen shock absorbers, and base frame (Fig. 10). The entire unit can be mounted either in a horizontal or vertical position.

The operation of the machine is simple; chamber A is charged with nitrogen gas to the desired firing pressure, and a trigger pressure is applied to the small area between chambers A and B. This trigger

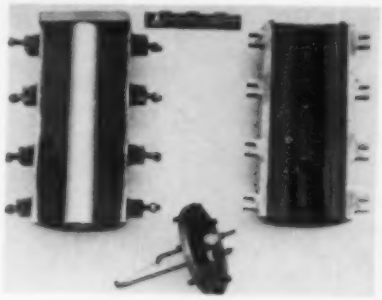
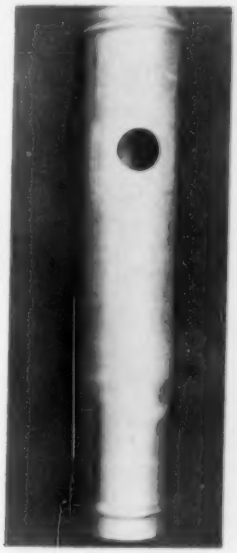


Fig. 8—Left, shows a split die, blank, and diehead electrode with initiating wire attached. Fig. 9—right, depicts the finished part.



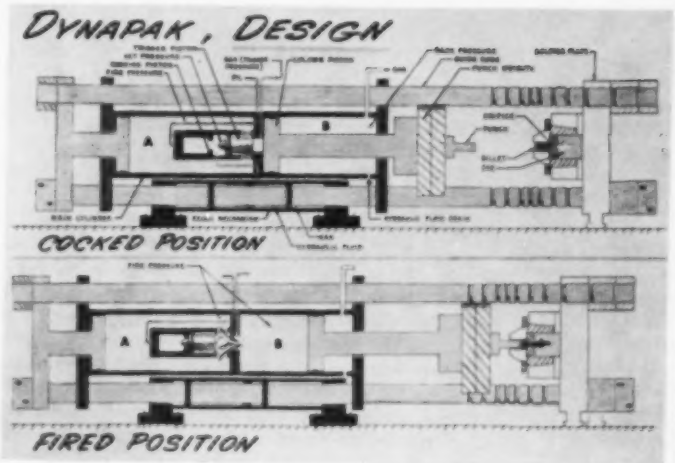


Fig. 10—Dynapak assembly is shown at the right. The entire unit can be used in either the horizontal or the vertical position.

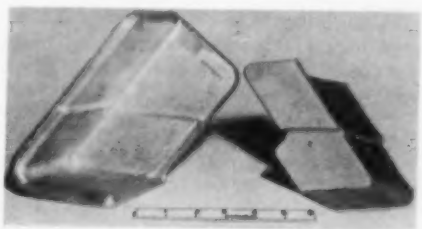


Fig. 11—Above, part at right was machined from the blocker forging shown at left. Fig. 12—Below, shows the results of producing the part with Dynapak.

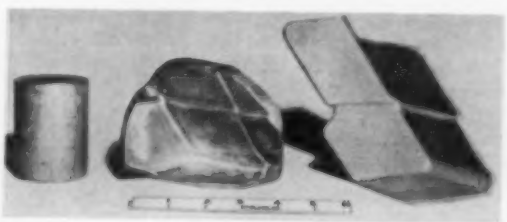


Fig. 13A—Below, illustrates a part of 19-9 DL stainless steel which was machined from a standard blocker forging.

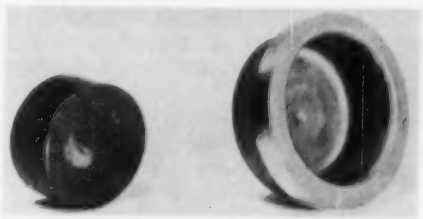


Fig. 13B—Below shows the part produced according to high energy rate principle.

Fig. 15—A comparison of a welded hook and flange (left) and a hook and flange using Dynapak.

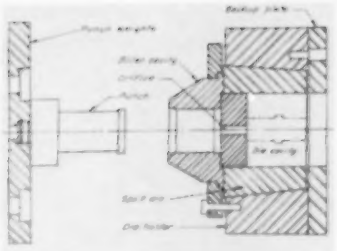


Fig. 14—Left, an illustration of the apparatus for closed-cavity extrusion molding.

pressure forces the trigger piston to an open position. Compressed nitrogen then passes through three large ports and acts upon the piston, driving it and the punch toward the work piece at a high velocity. The machine is mounted on a floating-type support; so, the bolster plate also moves toward the punch when the machine is fired. The momentum of these two cancel each other as the part is being formed; therefore, all stresses are contained within the machine. After the part is formed, the piston is returned to the cocked position by pumping hydraulic oil into the forward section of chamber B. This action also recompresses the nitrogen in chamber A to the original firing pressure.

Two forging firms in the Los Angeles area decided to give Dynapak a try. The Precision Forge Co. was the first, and its first customer, Convair-San Diego, ordered a forged gas-tight corner which would be structurally sound and require a minimum of machining. This part, shown on the right in Fig. 11, is used in the Convair 880 airliner. It was being machined from the 4-lb, 10-oz blocker forging (shown on the left in Fig. 11) to a finished weight of 10 oz. Fig. 12 shows this part as now produced finished forged from 2014 aluminum alloy in two strikes by Dynapak. The only machining required is the flash trim and the attach hole drilling. The change-over in production technique for producing the gas-tight corner resulted in considerable savings.

An example of what can be done with high energy rate applied to a part made from 19-9 DL stainless steel is shown in Figs. 13A and 13B. This part had been machined from typical blocker forgings, but Precision Forge Co. applied the high energy rate principle with a resulting 75 pct overall saving to the customer.

But die material selection and die design requirements have not been completely established, and continued development will be required to put this new process into full production.

Closed-Cavity extrusion molding

Improvements demonstrated in open die forging experiments conducted by Precision Forge Co. whetted the enthusiasm of Convair's engineer's to try the principle of closed-cavity extrusion molding, i.e. the forcing of metal in a semisolid or plastic state through a small orifice into a defined die cavity to produce a finished or semifinished configuration with good physical properties.

The initial work centered around the effect of moving metal through an orifice into a closed cavity. These tests were conducted using the die shown in Fig. 14 which was so designed that various orifice ratios could be employed.

The results of simple tests on 17-4 PH stainless steel and AISI 4130 steel indicated that these materials did experience the behavior of a fluid when subjected to the high energy rates. They also indicate that the metal, while no longer possessing defined longitudinal or transverse structure, did not suffer a loss of tensile properties. In fact, it now appeared to have equal longitudinal and transverse tensile strength.

Armed with this knowledge, the next step was to select some part of the B-58 airplane which offered a challenge, for example the canopy pod hook made of AISI 4130 and shown in Fig. 15. It is made of two pieces—flange and hook—which are welded together.

The die material selected was *Stentor* heat treated to a hardness of Rockwell C 55-60. After a series of tests, the results which are most dramatically portrayed in Fig. 15—with the original welded hook on the left and the Dynapak hook on the right—were achieved. All of these tests were not without great disappointment. During the fabrication of the next successful hook, the die failed. The failure can be attributed to a lack of knowledge of tool steels and the need for closer control of heat treating. Since this tool had to be rebuilt, several tool steel manufacturers were brought into the picture. They were all sympathetic and saw the need for a new die steel which is tough, abrasion resistant, thermal shock resistant, and has good elongation. With improved die steels, we believe that this process can literally cause complex parts to be born to shape.

Conclusions

The application of high energy rate to industrial-scale operations is feasible; however, there remains a considerable amount of development work still to be accomplished. It must be recognized that each method has its own area of application, and that no one method is the panacea for the materials processing industry.

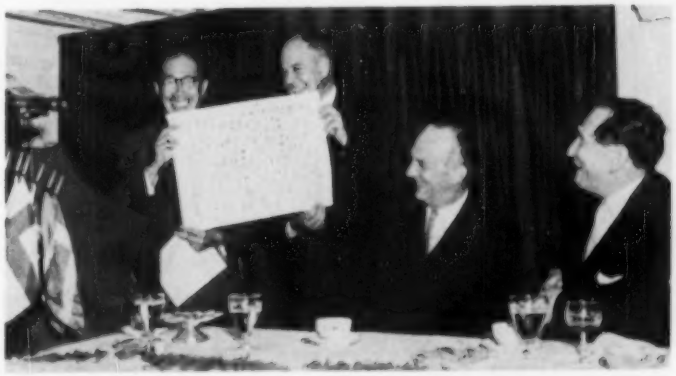
The several processes still require a great deal of manual work, and we must improve handling devices for tools and material. In addition, a whole new concept will be required in the design of quick opening dies. The design is made more difficult because of the high internal pressures acting upon the material and the dies.

With diligent effort on the part of industry, it is certain that the maximum utilization of high energy rate deformation will be made in the very near future. Through the application of these new processes, engineers are offered vast design freedom.

POWDER METALLURGISTS MEET

A Review of the International Powder Metallurgy Conference

June 13-17, New York City



A truly international symposium . . . Depicted above are (left to right) Y. Matsuyama of Japan, F. V. Lenel of the USA, and Ambassador Moch and P. Schwarzkopf both of Austria.

MORE than 18 months of detailed planning and intensive effort culminated in success when about 750 scientists and engineers, including 100 visitors from foreign countries, attended the first International Powder Metallurgy Conference to be held in this Country. The meeting, which was co-sponsored by The Metallurgical Society's IMD Powder Metallurgy Committee and the Metal Powder Industries Federation, convened at New York's Biltmore Hotel, June 13-17.

The proceedings got off to an auspicious beginning with a reception on Sunday evening in honor of the visitors from overseas—courtesy of an international assortment of industrial sponsors. Turning to serious business, conferees attended the first in a three-day series of technical sessions on Monday.

Prior to the start of the regular technical program, registrants were welcomed by the Conference Co-chairmen, F. V. Lenel of Rensselaer Polytechnic Institute; Kempton H. Roll, Metal Powder Industries Federation Executive Secretary; and Program Chairman C. G. Goetzel of New York University.

The keynote of the meeting was sounded by Paul Schwarzkopf, Honorary Chairman and President of Metallwerk Plansee GmbH. Reutte, Austria, who was introduced as *Mr. Powder Metallurgy*. In his talk entitled *Half a Century of Modern Powder Metallurgy*, Dr. Schwarzkopf outlined the highlights in the development of powder metallurgy technique and theory since 1910. He based his starting point on the successful application by Dr. W. D. Coolidge of powder metallurgy techniques to the problem of producing room-temperature-ductile tungsten for use in incandescent lamps. Citing prehistoric iron powder metallurgy and early 19th century production of sintered platinum in Britain and Russia, Dr. Schwarzkopf described 1910 as the year of renaissance in powder metallurgy.

The balance of the morning and the afternoon session of the first day of the Conference was devoted to a Symposium on Powder Metallurgy Fundamentals and Theory.

At 12:15, many conferees were to be found at the Welcoming Luncheon, which concluded with a short talk by K. H. Roll on *Powder Metallurgy and the Metal Powder Industry in the US and Canada*.

On Tuesday, two symposia were conducted dealing with Dispersion Strengthening and Powder Metallurgy Technology and Methods. Wednesday was occupied exclusively by a symposium on Powder Metallurgy Alloys, Materials, and Applications.

The international flavor of the Conference came to a definite peak at the Banquet, which was held on Tuesday evening starting at 7:30 pm. A congenial group of registrants and their wives enjoyed an evening program which left little doubt about powder metallurgy's worldwide scope. Following the introduction of those who were responsible for the planning and organization of the Conference, K. H. Roll relinquished the lectern to Co-chairman F. V. Lenel. Dr. Lenel introduced the following representatives of various countries whose scientific and professional organizations for powder metallurgy sent greetings on the occasion of the conference: Y. Matsuyama (Japan), B. A. Oliver (England), W. Hotop (Germany), and Michel Eudier (France).

Also attending the Banquet were representatives from Finland, Canada, and Sweden, and Karl Wolf, Consul-General of Austria in New York. Numerous duties next fell to the toastmaster, Carleton C. Long, 1960 President of The Metallurgical Society of AIME and director of research, St. Joseph Lead Co. Dr. Long read two letters of congratulations and greetings to those attending the Conference: the first was from George B. Kistiakowsky, scientific adviser to the President of the US; the other from Nelson A. Rockefeller, Governor of the State of New York. He then introduced His Excellency, Franz Matsch, the Austrian Ambassador to the United Nations. Dr. Long's final assignment before introducing the speaker of the evening was to present to the Honorary Chairman of the Conference, Paul Schwarzkopf, a plaque bearing the following inscription:

"Commemorating Half a Century of Modern Powder Metallurgy and Honoring a Pioneer in the Science and Art of Powder Metallurgy.

> Paul Schwarzkopf, Dr. of Tech. Sc. and Dr. of Philos. (Prague), Dr. h.c. (Graz), Senator h.c. and Prof. (Innsbruck), Baurat h.c.

Presented at the 1960 International Powder Metallurgy Conference, co-sponsored by the Metal Powder Industries Federation and the Institute of Metals Division, The Metallurgical Society of the American Institute of Mining, Metallurgical and Petroleum Engineers, June 14, 1960.

> Attached hereto is a specimen from one of the first sheets of coated molybdenum made by powder metallurgy under the direction of and by techniques, unique at the time, perfected by Dr. Schwarzkopf."

He then introduced Charles E. Reed, general manager, Chemical and Metallurgy div., General Electric Co., who spoke on "The Fifty Year History of Powder Metallurgy".

The last two days of the five-day meeting were devoted to plant tours offering conferees an opportunity to observe a variety of powder metallurgy operations ranging from metal powder production to fabrication and utilization of powder metal parts. Plants visited during the two-day period included Hoeganaes Sponge Iron Corp., Riverton, N. J.; F. J. Stokes Corp., Philadelphia, Pa.; Presmet Corp., Worcester, Mass.; Lionel Corp., Irvington, N. J.; Alan Wood Steel Co., Conshohocken, Pa.; Pitney-Bowes, Inc., Stamford, Conn.; and Stevens Institute of Technology, Hoboken, N. J.

Among those responsible for the planning and smooth operation of the conference, in addition to persons mentioned above, were: H. H. Hausner, Consultant (international liaison), R. W. Shearman, Secretary, The Metallurgical Society of AIME (AIME liaison), J. L. Bonanno, Lionel Corp. (Plant tours), Chalmers Dale, Charles Hardy, Inc. (Social activities), and Werner Leszynski, Schwarzkopf Development Corp. (editorial).

All of the papers presented at the Conference, as well as written discussions of the papers, are to be published in a Proceedings volume by Interscience Publishers of New York.

The Technical Sessions

JOURNAL OF METALS *wishes to thank L. L. Seigle of General Telephone and Electronics Laboratories and F. N. Rhines of the University of Florida for providing reviews of technical sessions of the International Powder Metallurgy Conference.*

Powder Metallurgy Fundamentals and Theory

Reviewed by L. L. Seigle, General Telephone and Electronics Laboratories.

In the morning session, a group of papers were presented dealing with the theory of sintering. These were introduced by George C. Kuczynski of the University of Notre Dame, with a review of his classical work on the fundamentals of mass transport during sintering. In addition to reviewing the present status of the theory, Dr. Kuczynski in his paper, *Theory of Sintering*, discussed his recent work on the sintering of aluminum oxide single-crystal spheres and the striking effect of atmosphere on the mass transport processes involved in the bonding of such spheres. *Sintering Behavior of Organic Materials* by G. A. Geach and A. A. Woolf, of Associated Electrical Industries, Ltd., England; *Sintering Mechanism of Titania* by G. Parravano, H. M. O'Bryan, Jr., and V. J. Lee, of the University of Michigan; and *Sintering of Wire Compacts* by A. L. Pranatis and L. L. Seigle of General Telephone & Electronics Laboratories Inc., brought to bear an interesting range of experimental data on the fundamental mechanisms of sintering proposed by Kuczynski and his co-workers.

Sintering Behavior of Organic Materials

by G. A. Geach and A. A. Woolf, Associated Electrical Industries, (Manchester) Ltd., England.

In studies of the sintering of a range of organic molecules and one organo-metallic compound, strong confirmation was obtained of the fact that diffusional, rather than creep, processes are essential to sintering of powders of such substances. Particles of these organic compounds with high molecular weight, in which diffusion is extremely slow, underwent no sintering, even at temperatures about 75 pct of their absolute melting points. The substances which were examined underwent creep at these temperatures. Hence, it is concluded that diffusional processes are essential to particle bonding in these materials, and plastic flow does not contribute to sintering.

Sintering Mechanism of Titania

by G. Parravano, H. M. O'Bryan, Jr., and V. J. Lee, University of Michigan.

Studies by Parravano and co-workers indicate on the other hand, that sintering in titania single-crystal microspheres apparently occurs by a plastic flow mechanism in the temperature range of 900° to 1350°C. This was indicated by a kinetic analysis of the neck growth data. The authors claim that under the experimental conditions used in their research, the sintering of titania did not involve a diffusional mechanism but followed a behavior different from that previously found for zinc and other oxides. Neck formation under certain conditions occurred by the growth of a bridge of whiskers between spheres.

Sintering of Wire Compacts

by A. L. Pranatis and L. L. Seigle, General Telephone and Electronics Laboratories, Inc.

Careful studies of the sintering of nickel, copper, and iron wire compacts by A. L. Pranatis and L. L. Seigle confirmed that bonding between wires involved a volume diffusion mechanism in nickel and copper at temperatures near the melting points, and a surface diffusion mechanism in iron compacts at about 0.6 of the melting point. These results support the Kuczynski theory. Data on the elimination of voids in these wire compacts did not fully confirm previous results regarding the influence of grain

A pioneer in the field . . . Society president C. C. Long presents Dr. P. Schwarzkopf with a plaque commemorating a half century of endeavor in powder metallurgy.

boundaries on the process of sintering. Grain boundaries not only seemed to have no influence whatever on the rate of neck growth between wires, but had varying effects on the rates of shrinkage. This is in contradiction to previous results. In the discussion period, Kuczynski presented evidence that grain boundaries did not affect both neck growth and rate of densification. This was followed by a lively debate on various controversial points.

Interparticle Contacts in Sintering
by F. N. Rhines and R. T. DeHoff, of the University of Florida.

The afternoon session of the Symposium was concerned with other aspects of powder metallurgy fundamentals and theory. F. N. Rhines and R. T. DeHoff discussed the geometrical factors important in sintering. Both theoretical and experimental evidence was presented showing that the rate of densification is proportional to the number of contacts, and the number of contacts remains constant during the first stage of sintering.

Contribution to Liquid Phase Sintering
by F. Eisenkolb, Technische Hochschule, Dresden, Germany.

Sintering in the presence of a liquid phase was discussed by F. Eisenkolb who emphasized the theoretical and practical importance of the introduction of liquid phases in accelerating solid-state reactions during sintering. Data were presented on the results of additions of metallic and non-metallic fluxes and slags to pure and alloyed iron powders. The effects of these additions on dimensional changes and mechanical properties were discussed. The practical utility of sintered powders containing additions of sulfides or silicates was a major point of discussion.

Special Type of Activated Sintering
by J. Wulff of the Massachusetts Institute of Technology.

Dr. Wulff pointed out that it has been known for some time that the rate of densification of a metal powder can be markedly accelerated by the addition of a second metal with a higher coefficent of self-diffusion. This device can be employed to produce sintered bodies of refractory metals, such as tungsten, with densities greater than 95 pct of theoretical at temperatures appreciably below those otherwise required.

Sintering with a Chemical Reaction as Applied to Uranium Monocarbide
by R. Caillat, A. Accary and I. Duchuissan of the French Atomic Energy Commission.

As stated by R. Caillat and co-workers, special techniques are essential in preparing uranium carbide bodies for nuclear reactors. Cold pressing and conventional sintering does not result in a highly dense part, but satisfactory results can be obtained by hot-pressing mixtures of uranium and graphite particles and allowing the uranium carbide to form by chemical reaction. A simultaneous application of heat and pressure is essential.

A Plastic Flow Model of Hot Pressing
by J. D. McClelland of Atomics International.

Mr. McClelland discussed the theory of hot pressing. He showed that the rate of densification of beryllia compacts under pressure as consistent with the theory of Mackenzie and Shuttleworth. This shows that the principal driving force for the closing of pores during hot pressing is the applied hydrostatic pressure, and the mass transport mechanism is plastic flow, as expected.

High Density Sintering of Metal Powder Compacts
by M. Eudier, Metallurgie des Poudres, France.

Mr. Eudier reviewed the different conventional ways of obtaining high-density parts, including high-temperature sintering, hot pressing, hot forging, rolling, etc. He further discussed other possibilities, especially the use of ultra-fine powders, special atmospheres on thin films, and intermediate liquid phases created by small additions such as phosphorus, which can later be eliminated.

Microhardness Testing in Powder Metallurgy Research
by H. Bückle, Office National d'Etudes et de Recherches Aeronautique, France.

The final contribution to the Symposium was made by H. Bückle who showed that a comparative study of hardness variations can be used to obtain valuable information about the structure of heterogeneous or porous bodies. With proper interpretation of the variation of microhardness values with load, the intrinsic hardness of dispersed phases in a matrix can be established. Such measurements also can supply a non-destructive determination of the thickness and nature of thin protective coatings.

The paper *Sintering of Very Fine Oxide Particles Produced by Calcination of Salts* by K. Iwase, T. Takada and T. Hayashi, of the Kyoto University, Japan, was not presented due to the absence of the authors, but the full text will appear in the proceedings of the conference.

Dispersion Strengthening

Reviewed by F. N. Rhines, head professor of the Metallurgical Research Lab.—the University of Florida.

The Role of Powder Metallurgy in Dispersion-Strengthened Alloy Development
by C. G. Goetzel and R. F. Bunshah, New York University.

Applications of the SAP method of hardening metals by the incorporation of a fine dispersoid are beginning to focus attention upon nickel, iron, and copper as carrier metals and upon the highly refractory oxides as dispersoids. Goetzel and Bunshah, in reviewing the combinations that have been tried and the production methods that have been developed, pointed out that fineness of the dispersoids is of the greatest importance. Strength and hardness at high temperature appear to result not only from the slip interference afforded by the second-phase particles, but also, and to a greater degree, from the retardation of the recrystallization and grain growth caused by the presence of the dispersoid. When fine particles become agglomerated by long holding at high temperature, they lose their capacity to prevent grain growth. This holds true even after subsequent cold working.

Properties of Nickel Containing Dispersed ThO_2
by D. K. Worn of Mond Nickel Co. Ltd. and S. F. Marton of Renold Chains Ltd.

The foregoing paper was enlarged upon by Worn and Marton, whose studies upon nickel hardened with thoria revealed that the agglomerating tendency can be controlled to some extent by controlling the impurity content of the alloy. Similar effects were reported by the Japanese workers, Y. Imai and H. Hirotani of Tohoku University in their paper, *Dispersion Strengthened Steels, Particularly Heat Resistant Steels*. Their studies centered on the ferritic and austenitic stainless steels hardened with alumina, silica, and magnesia.

Latest Developments In The SAP Process
by E. A. Block and H. Hug, Aluminum-Industrie A. G., Switzerland.

Progress with SAP itself—aluminum hardened with its own oxide—as reported by Block and Hug was most noteworthy in the field of new applications. Many kinds of machine parts, which require mechanical stability at high temperature, have come into use.

Internal Oxidation of Dilute Solutions of Ni and Cu for Dispersion-Strengthened Metal-Metal Oxide Alloys
by N. Komatsu, L. J. Bonis, and N. J. Grant, Massachusetts Institute of Technology.

The Meijering technique of producing a hardening dispersoid by internal oxidation has been the subject of a highly successful study by Grant and his co-workers. Mechanical properties were studied both in alloy wires, in which an oxide dispersoid was produced by internal oxidation subsequent to the forming of the wire, and in sintered alloy powders, in which the dispersoid was developed by interaction between the powder and the atmosphere. The most promising alloy systems for this application are those in which the oxide that forms has high thermodynamic stability, since the finest particles are produced by the internal oxidation of such alloys. A problem of considerable difficulty to which Grant and co-workers addressed themselves was that of avoiding oxide segregation in the sintered powder materials. In this area, control of the quantity of oxidizing element in the alloy is most important.

A Theory of Dispersion Strengthening
by F. V. Lenel and G. S. Ansell, Rensselaer Polytechnic Institute.

Lenel and Ansell proposed a new theory to account for hardening by dispersoids. The prior theory postulates that a dislocation, in order to pass a particle, must form a closed loop about the particle and then break away. The energy of hardening is derived from the dislocation loop formation. The new theory proposes that the dispersoid particle actually shears (or fractures) to permit the dislocation to pass, and that the energy of hardening is that energy required to shear the particle in the path of a moving dislocation. On this bases, the effect of particle size on the proportional limit was predicted and was shown to conform with experiments.

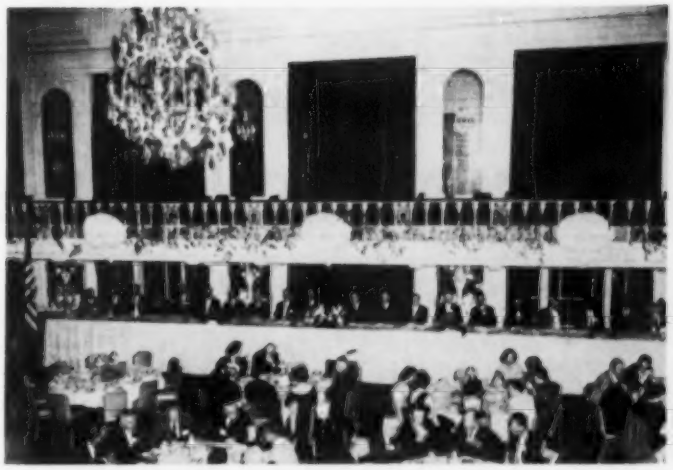
Roll Forming of Metal Powders
by D. K. Pickens, Chemetals Corp.

The important methods of producing finished parts by powder metallurgical techniques were reviewed. Pickens outlined the history of roll forming of metallic and non-metallic powders, and described, in detail, production equipment currently in use. The development of a newly designed rolling mill was of particular interest. The mill consists of two large rings, driven by smaller rolls adjacent to the area of pressure application. The rings serve as rolling surfaces, thus providing a radius of curvature of nearly 6 ft at the pressure points and permitting greatly increased rolling pressures. In a brief discussion of this work, a technique for hot rolling of powders was described. Roll-formed powder strips showed superior strength and ductility, in addition to favorable costs, when compared to conventionally-produced powder strips.

Hot Extrusion of Metal Powders
by P. Lowenstein, L. Aronin and A. Geary, Nuclear Metals Inc.

Lowenstein et al. described several techniques for the hot extrusion of metal powders. Particular emphasis was placed upon the practice of extruding a powder held in a metal can. This technique permits extrusion of toxic and pyrophoric powders into a wide variety of shapes and sizes. The effects of such variables as temperature, reduction ratio, particle size, and die geometry were briefly discussed.

Hydrostatic Pressing of Metal Powders
by H. Hirsch and G. E. Van Buren, General Electric Co.



Banquet . . . . . Powder metallurgists and their wives take part in a fine meal in an elegant setting.

The formation of powder compacts by hydrostatic pressing was discussed by Hirsch and Van Buren. The general technique was outlined and typical equipment was described. It was found that de-airing the charge has little effect on the resulting compact, and that compaction pressures above a certain minimum which is characteristic of the material result in little change in the properties of the finished part. The process may have great advantages for large parts, but it is currently not applicable to mass production of small parts. The versatility of the process—a wide variety of shapes can be produced—was emphasized.

Slip Casting of Metal Powders and Metal-Ceramic Combinations
by H. H. Hausner, Consultant, and A. Poster, Sylvania Electric Products Inc.

Hausner and Poster described the process of slip casting metal powders. A correlation was found between the pH of the slip, its viscosity, and the green density of the resulting casting. A maximum green density was obtained from slips having minimum viscosity. The properties of slip-cast and sintered parts compare favorably with those of pressure-compacted powders.

Explosive Forming of Metal Powders
by R. S. Cooley, Propellex div., Chromalloy Corp.

The explosive forming of metal parts was described by Cooley with emphasis upon the variety of applications possible. Application of the process to the compaction of metal powders was briefly described. The process permits the attainment of high hardness levels in powder parts without large dimensional change.

A detailed summary of explosive forming techniques by John Pearson of the US Naval Ordnance Test Station at China Lake, Calif., is printed in this issue of the JOURNAL OF METALS.

Lubricants for Powder Metallurgy Parts Manufacturing and Their Influence in Properties
by E. J. Geijer, Hoeganaes Sponge Iron Corp.

Geijer explored the effect of varying the amounts of several lubricants upon the green density and stripping pressure and the sintered properties of several metal powders. The type of lubricant used in pressing was found to have very little influence upon the density and strippng pressure of the compact form. However, these properties vary markedly with the amount of lubricant used. On the other hand, properties of the sintered compacts, such as hardness and strength, were found to be sensitive to both variables.

Large Sintered Bodies and Parts
by F. Emley, Westinghouse Electric Corp.

Emley graphically illustrated that most of the foregoing methods have no size barrier. Very large parts have been pressed and sintered and size limits upon most compacting processes are not yet in sight. Powder mteallurgy has great economic advantages in the fabrication of larger sized parts.

Wednesday sessions

On Wednesday the Symposium on Powder Metallurgy Alloy, Materials, and Applications brought forth a number of outstanding papers, and JOURNAL OF METALS regrets that it was unable to arrange for coverage of this session. However, two or three of the best of these papers will be published in full in a forthcoming issue.

Conclusions

The 20th century has seen the powder metallurgy industry grow from infancy to a state approaching maturity. Research activities of the last few decades have culminated in such processes as continuous powder rolling, slip casting, and indirect sintering of large tungsten and molybdenum ingots—in addition to the production of cermets and SAP materials.

Mr. Powder Metallurgy, Paul Schwarzkopf, presented a clear picture of the need for continued research in his keynote address. He stated that " . . . during the last 50 years, we answered quite a few questions and solved quite a few problems. But there are many unanswered questions left. Some of these will . . . be answered in the course of this conference, but the number of questions left will not become smaller. A wise man once said that knowledge is like a sphere—the larger the sphere, the more are the points of contact with the unknown."

The International Powder Metallurgy Conference did provide many answers to important questions and the sphere became larger.

MAX PLANCK INSTITUTES FOR METALLURGICAL RESEARCH

This is another article in the JOURNAL OF METALS' *series on important European metallurgical research centers. Previously published articles dealt with the Belgian National Metallurgical Research Center, CNRM, and the British Iron and Steel Research Association, BISRA.*

1. The Max-Planck-Institut für Eisenforschung*, · Düsseldorf, Germany.

by W. Wepner

The Max-Planck Institut für Eisenforschung (ferrous research) in Düsseldorf, Germany, was founded in June 1917 by representatives of the German iron and steel industry, called together by the Verein Deutscher Eisenhüttenleute. The Institute was later joined with the Kaiser-Wilhelm Society—today the Max-Planck Society for the Advancement of Science—in order to ensure freedom in scientific matters from outside interest groups.

W. WEPNER is a member of the scientific staff of the Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany.

* The Max-Planck Institute for Ferrous Research.

The area of the Institute's work encompasses basic and applied research in the chemical and physical metallurgy of iron and steel production, shaping, and forming. A practical point of view is achieved by the work of the Institute's scientists and engineers with the divisional committees of the Verein Deutscher Eisenhüttenleute.

The Institute in Düsseldorf is divided into a series of laboratories whose work is generally either self-contained or carried out in conjunction with other laboratories of the organization. Activities are carried on for other Max-Planck laboratories on an exchange basis, according to the special facilities

available in each particular unit, e.g. chemical analyses, microscopic research, and mechanical testing. Finally, cooperative work is carried out with the technical committees of the Verein Deutscher Eisenhüttenleute and the individual plants. Results of the work is—in accordance with the charter of the individual Institute—made available through publication. At present, some 280 persons—of whom 60 are scientists and engineers—are employed at the Eisenforschung Institut.

Research program

While the general objectives of the Institute have remained unaltered since its founding, the emphasis in certain fields has changed in many ways. In the last 15 years, physical metallurgy has moved forward, and physical methods of measurement have been used increasingly. It is impossible to review all of the Institute's work here; so, only projects to be completed shortly or still underway shall be mentioned.

For most basic research work, the purest iron possible is desired as a raw material. In addition to determining the characteristics of pure iron and its changes through minute alloying, research on the production of very pure iron in high vacuum has been performed. Investigation of further purification through zone melting is underway.

The steel worker, of course, has a constant interest in the fastest possible analysis procedure to control the various steelmaking processes. A group in the chemical laboratories is studying improvements of known analysis techniques and the development of new ones in order that the speed of the procedure may be made as rapid as possible.

For a number of years, the electrolytic isolation technique has been used for chemical-metallurgical research. The Institute examined experimental conditions in detail and several techniques were developed to separate the constituent parts of the isolated phases. The deposit is examined micro-chemically, with X-ray and electron microscope. Electrochemical work is concerned with stress corrosion, particularly in steel reinforcing rods, as well as with pit corrosion.

Mechanical working

Mechanical working processes—cold and hot rolling, deep drawing (or cupping), wire drawing, rod drawing, and ramming—are the object of exhaustive research. Recently the question of the suitability of various greases, rolling oils, and rolling oil emulsions was investigated. A study of wear on drawing tools was carried through with the help of some drawing tools that, by irradiation in an atomic pile, had become radioactive. Surfaces of pulled rods picked up particles from the drawing blocks which could be measured with a scintillation counter.

An extensive research project concerning the mechanical and electrical power supplies of cold- and hot-rolling equipment under operating conditions is being carried out in various rolling mills. The rolling mill technical organization of the Verein Deutscher Eisenhüttenleute, as well as firms producing rolling mills and electrical driving mechanisms, are cooperating in this venture.

Fracture

For the application of steels to construction purposes, behavior under multi-axial stress and susceptibility to brittle fracture, particularly at low temperatures, is being examined. Typical tests include tensile tests on tubular pieces under equilateral internal pressure. Extensive creep-rupture experiments, continuing over several years, should clarify the behavior of ferritic and austenitic steels by continual stress at raised temperatures. Structural changes are observed microscopically, electromicroscopically, and with the aid of electrolytic isolation techniques.

The behavior of steels under alternating or vibrating stresses is examined through continual vibrating tests at various temperatures—again using combined metallographic and chemical techniques.

Transformation studies

For many years work has continued on transformation characteristics and transformation kinetics, segregation incidence, and segregation kinetics of steels. Extensive dilatometric measurements were the basis for drawing up TTT diagrams; these diagrams, drawn up for standard German steels, were published under the title, *Atlas zur Wärmebehandlung der Stähle.*

The time factor in transformation and segregation is determined by various methods, including magnetic susceptibility, measurements of internal friction, and micro-calorimetric measurements. By the latter technique, the small quantity of time-dependent heat given forth is measured. For that purpose, water-vapor thermostats under the constant pressure of boiling water were built and erected in an air-conditioned room, thereby making a very exact reference temperature possible. In the interior of the thermostats, the metal block, containing the measuring cell, is maintained at the test temperature with the aid of regulated direct current. Through these arrangements, measurements of the precision of a thousandth part of a degree per day are reached. The heat produced is thermoelectrically measured with a differential technique and recorded.

In the field of non-destructive testing, the evaluation of indicated flaws by irradiation and the identification of flaws as influenced by test conditions, is investigated by X-ray. Betatron-produced X-ray and radioactive isotopes are also used in this work.

Atomic structure

Atomic structural research utilizing X-rays is concentrated on the strain and distortion phase in polycrystalline samples. For these tests, the most exact measurements of X-ray line intensity distribution is necessary. As a consequence, an X-ray geniometer with a scintillation counter and a differential discriminator was established:; this system is superior to the formerly-used photographic methods and Geiger-Müller counters. Electron and neutron diffraction techniques are also used for structural analysis.

The neutron spectrometer is temporarily located at the Belgian atomic center in Mol. But it will be placed in the soon-to-be-completed atomic research unit in North—Rhine Westphalia in Germany.

2.

The Max-Planck-Institut für Metallforschung,* Stuttgart, Germany.

The Special-Metals Division of the Max-Planck-Institute für Metallforschung.

by Werner Koster

The establishment of an institute for metallurgical research within the framework of the Kaiser-Wilhelm-Gesellschaft zur Förderung der Wissenschaften (Kaiser-Wilhelm-Society for the Advancement of Science), the predecessor of the present Max-Planck-Gesellschaft (Max Planck Society), was initiated before World War I. However, the realization of these plans became possible only several years after the end of that war.

The history of the former Kaiser-Wilhelm-Institut für Metallforschung and the present Max-Planck-Institut für Metallforschung (Max Planck Institute for Metallurgical Research) may be divided into three periods. The first period began with the opening of the Institute in Berlin-Neubabelsberg in the year 1921. In the following year, shortly after the death of its first director, Emil Heyn, financial consideration forced the Institute to move into another suburb of Berlin; here it remained until 1933. During those early years of its existence, operations were supported exclusively by funds contributed by the German metals industry. But, again in 1933, financial difficulty forced the research establishment to close temporarily. Thanks to the joint efforts of industry, science, and the public, the Institute was re-established the following year—this time in Stuttgart. The opportunity to combine the new research facilities with the existing laboratories of the Stuttgart Technische Hochschule (Stuttgart Technical College) played an important role in the relocation.

The Stuttgart Institute originally embraced three constituent institutes: Institut für Metallkunde (Institute for Metallurgy), Institut für physikalische Chemie der Metalle (Institute for Physical Chemistry of Metals), and Institut für Metallphysik (Institute for Physical Metallurgy). The leaders of these departments were associated with the Technische Hochschule and this close co-operation remains characteristic today, with four of the five leaders in charge of the constituent institutes holding posts at the Hochschule and numerous students performing their scientific work in the laboratories of the Institute.

Post war developments

Considering the serious losses sustained by the Institute during and after World War II-in which most of the buildings and equipment were destroyed—the constant growth since its re-opening is truly remarkable. The number of persons comprising the permanent research staff increased from the pre-war 10 to the present 25.

Although specific research projects and methods changed with time, the Institut's basic purpose has remained the same throughout its 40-year existence.

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* The Max-Planck Institute for Metallurgical Research.

That purpose encompasses the scientific research of the chemical and physical properties of metals, together with investigations into methods of metal extraction and working. Emphasis centers mainly, but not exclusively, on nonferrous metals. Within the limitations of this article one can only mention several examples in order to give an impression of the variety of fields and goals with which the Institute has associated itself through the years.

Structural studies

Research into alloy constituents has played a prominent role. In the beginning the main interest lay in binary systems, but later it was transferred to three or more constituent systems. With the help of constantly improving methods of physical measurement, research was concentrated on the heat treatment of alloys—where precipitation hardening commanded the greatest attention. In the early days, detailed studies were conducted on the nature and kinetics of cold-precipitating aluminum alloys. These studies are outstanding examples of the close co-operation between the various research groups of the Institute. The typical metallurgical investigations were supplemented with extensive studies by X-ray techniques on the structure of Guinier-Preston zones, which originate in the process of cold-precipitation hardening. X-rays were used to investigate other theoretical-physical phenomena to learn more about the thermodynamics and the atomic structural changes of the underlying basic mechanisms. In this connection, recent studies (with the help of electrical resistance and Hall-effect measurement) on the phase transformation of several alloys should be mentioned.

Structural studies, based on X-ray and electron bending techniques to describe alloy phases have been conducted for years. Through this project, deviation from the regular crystal structure in form of discontinuities or the so-called dislocations became especially interesting. Among structural studies, the investigations on solid-amorphous materials and metal slags should also be mentioned.

Metal physics

Intense interest has also been shown in the field of metal physics, and its growing significance prompted extensive enlargement of research facilities during the last decade. Theoretical investigations using electrons (the beginnings of which can be traced back to the 1930's) to understand the binding and structural relations of metals recently laid emphasis on the phenomena of ferromagnetism and transport processes. The decisive influence of lattice imperfections was recognized early, and many theoretical and experimental works made substantial contributions to the knowledge of lattice imperfections and their influence on different metal characteristics.

The following can be mentioned as examples of the application of theory to practice: the displacement theory of form-hardened crystals, the interpretation of radiation damage, and the influence of lattice imperfections on the ferromagnetic properties. Discontinuity effects contributed to the understanding of precipitation and transformation processes—as in the case of cobalt transformation, martensite formation, and nucleation in the solid phase.

To illustrate the variety of investigations on the properties of metals and alloys, further examples may be cited: study of relation phenomena by measurement of mechanical damping, investigation of elastic constants and their dependance on temperature, and projects concerning technical properties and surface protection of different alloys.

Magnetic properties

In recent years, considerable time has been spent in studying the relation between structure and magnetic properties, resulting in the knowledge of the permanent magnetism of alloys. This work precipitated the investigations of the relationship of residual stress to the shape of the magnetization curve. It has lately been concentrated on the influence that dislocations exert on magnetic properties. These studies serve as a starting point for the interpretation of the magnetic phenomena on the basis of the strain hardening mechanism, and contributed to the explanation—likewise developed in the Institute—of residual stress through magnetic properties. These studies produced the interpretation of magnetic phenomena with the help of the strain-hardening mechanism; furthermore, they contributed to the theory (Institute developed), explaining residual stress through displacement processes. Extensive attention also has been given to the influence of gases and their effect on the properties of metals.

Plasticity

Another field cultivated since the founding of the Institute is the shaping and forming of metals. This work—still in Berlin—on the plasticity of single crystals resulted in the classic findings on the deformation mechanism. Recent investigations by means of the displacement theory and precise experimental methods, such as the electronmicroscopic investigations of surface phenomena, succeeded in providing an understanding of the deformation process in simple crystal structures.

In order to clarify the deformation in multiple crystals, measurements on the deformation and residual stresses of the single crystallite are in progress. The X-ray methods used for this purpose are based on the process developed by the Max-Planck-Institut and applied to many problems throughout the years.

Other studies

In summary we may mention the studies of; viscosity and conductivity of molten metals, crystal growth and development in the microstructure, stress corrosion, and electrochemical phenomena of metallic electrodes. Likewise, it is impossible to go into details about the work on the dosimetry of ionizing radiations which originated in related problems of X-ray application, investigated by Stuttgart for the past several years with remarkable results.

In view of the increasing importance of the special metals—especially those used in nuclear power reactors—a new department was set up to investigate their properties. The research of special metals will thus also bring the Max-Planck-Institut into this important field of metal science.

SOVIET METALLURGICAL AND CERAMIC JOURNALS -- A Selected List

Compiled by J. W. Corbett and J. B. Newkirk

The scientific and technical literature of the USSR is experiencing an explosive growth at least equal in rate to that occurring outside the Soviet sphere. Moreover, there has been an increased flow of Soviet publications to the United States. As a result, a bewildering number of journals, published in the Soviet Union, are now available in this country.

This paper is essentially a compilation of the titles of metallurgical and ceramic journals that currently are available in the US, and which may be of interest to scientific or engineering personnel. Specifically excluded from the list are serial publications, which appear at irregular intervals and are not classified as journals.

In this list the titles are arranged alphabetically according to the English alphabet. Each entry includes, besides the transliterated title, a) the title of the journal in English; b) the frequency of issue; c) the current cost per year of the journal in the original language; d) an identification of the original language of the journal; e) an abbreviation designating the translating agency, if any; and f) the cost-per-year of the translation subscription.

About 72 technical journals in the languages of the Soviet Union (mostly Russian) are currently being translated from cover to cover. Of these 72, 11 are of sections of *Doklady Akademii Nauk SSSR*, four are of sections of *Izvestia Akademii Nauk SSSR*, and two are sections of *Referativny Zhurnal*. Thus, a total of about 55 separate Soviet journals is regularly being made available in English. The delay between an original issue and its translation varies widely, but is in general from six months to a year. The key to translating agencies includes the address of the agency. Subscriptions to the translation may be placed directly with the agency.

We believe this list of metallurgical and ceramic journals is as complete and accurate as is practical, consistent with its being usefully brief. Published sources of Soviet science literature are themselves often incomplete, inaccurate, and contradictory. It should also be borne in mind that the information in this article is subject to change. Nonetheless, the listings should be helpful as a general guide.

Language symbols

Non-Russian journals sometimes have Russian language abstracts; This is indicated by an *R* after the primary language symbol.

R	Russian
Ar	Armenian
Az	Azerbaidzhan
B	Belorussian
G	Georgian
L	Latvian
U	Ukrainian

Translation agencies symbols

AIP	American Institute of Physics, 335 E. 45th St., New York 17, N. Y.
BISI	The Secretary, Iron and Steel Institute, 4 Grosvenor Gardens, London, S.W. 1, England.
BWRA	British Welding Research Assn., 29 Park Crescent, London, W. 1, England.
CB	Consultants Bureau, 227 W. 17th St., New York 11, N. Y.
HB	Henry Brutcher, Technical Translations, P.O. Box 157, Altadena, Calif.
ISA	Instrument Society of America, 313 Sixth Ave., Pittsburgh 22, Pa.
P	Pergamon Press, 122 E. 55th St., New York 22, N. Y.
PERA	Production Engineering Research Assn. of Great Britain, Stavely Lodge, Melton Mowbray, Leicestershire, England

Original language journal subscription agencies

Subscriptions for the journals in the ORIGINAL language can be placed with the following agencies:

1. Victor Kamin, Inc.,
   2906 14th St., N.W.,
   Washington 9, D. C.

J. W. CORBETT and J. B. NEWKIRK are with the General Electric Research Laboratory, Schenectady, N. Y.

2. Imported Publications and Products,
4 W. 16th St.,
New York 11, N. Y.

3. Four Continent Book Corp.,
822 Broadway,
New York 3, N. Y.

4. Cross World Books and Periodicals
56 E. Chicago Ave.,
Chicago 11, Illinois

5. Collett's Holdings Ltd.
44 & 45 Museum St.,
London, W.C. 1, England

Journal	Language	Translating Agency	Translation Subscription (cost per year)	Issues Per Year	Original Journal (cost per year, $
Atomnaia Energiia	R	CB	$75.00	12	14.50
Atomic Energy					
Prilozhenie k "Atomnaia Energiia"	R				
Supplement to "Atomic Energy"					
Avtomaticheskaia Svarka	R	BWRA	£10.10.0		
Automatic Welding					
Biulleten' Izobretenii	R	P	$100.00	24	8.00
Bulletin of Inventions					
Doklady Akademii Nauk Armian. SSR	Ar			12	6.00
Reports of the Academy of Sciences of the Armenian SSR	R				
Doklady Akademii Nauk Azerb. SSR	Az			12	5.00
Reports of the Academy of Sciences of the Azerbaidzhan SSR	R				
Doklady Akademii Nauk Belo. SSR	R			12	2.80
Reports of the Academy of Sciences of the Belorussian SSR					
Doklady Akademii Nauk SSSR	R			36	70.00
Reports of the Academy of Sciences of the USSR					
Crystallography Section					
Mechanics Section					
Theory of Elasticity					
Technical Physics		CB	$200.00		
Doklady Akademii Nauk Uzb. SSR	R			12	2.30
Reports of the Academy of Sciences of the Uzbek SSR					
Dopovidi Akademii Nauk Ukr. RSR	Ukr			12	9.00
Reports of the Academy of Sciences of the Ukrainian SSR	R				
Fizika Metallov i Metallovedenie	R	P	$30.00	12	20.00
Physics of Metals and Metallurgy					
Fizika Tverdogo Tela	R	AIP	$55.00	12	30.00
Solid State Physics					
Izobretatel i Ratsionalizator	R			12	6.00
Inventor and Innovator					
Izvestia Akademii Nauk Armian. SSR	Ar				
Bulletin of Academy of Sciences of the Armenian SSR	R				
Serila: Tekhnicheskikh Nauk					
Technical Sciences					
Izvestia Akademii Nauk Azerb. SSR	Az			6	6.00
Bulletin of the Academy of Sciences of the Azerbaidzhan SSR	R				
Izvestia Akademii Nauk Belo. SSR	B			4	4.00
Bulletin of the Academy of Sciences of Belorussian SSR	R				
Izvestia Akademii Nauk SSSR	R			12	20.00
Bulletin of the Academy of Sciences USSR					
Otdelenie Tekhnicheskikh Nauk					
Section of Technical Sciences					
Izvestia Akademii Nauk Turkmen. SSR	R			6	4.00
Journal of the Academy of Sciences of Turkmen SSR					
Izvestia Akademii Nauk Uzb. SSR					
Bulletin Academy of Sciences of Uzbek SSR					
Serila: Tekhnicheskikh Nauk	R			6	4.00
Technical Sciences					
Izvestia Karelskogo i Kolskogo Filialov Akademii Nauk SSSR	R			4	4.00
Bulletin of the Karelian and Kalo Peninsula Branches of the Academy of Sciences of the USSR					
Izvestia Sibirskogo Otd. Akademii Nauk SSSR	R			12	11.00
Bulletin of the Siberian Branches of the USSR Academy of Sciences					
Izvestia Vyshikh Uchebnykh Zavedenii	R				
Report of Universities of the Ministry of Higher Education					
Chernaia Metallurgiia				12	16.00
Steel and Iron Metallurgy					
Gornyi Zhurnal				12	12.00
Mining Journal					
Tsvetnaia Metallurgiia				6	8.50
Nonferrous Metallurgy					
Kislorod	R			6	4.00
Oxygen					
Kristallografiia	R	AIP	$25.00	6	12.00
Crystallography					
Kuznechno-Stampovochnoe	R			12	8.00
Forging and Stamping Industry					
Latvijas PSR Zinatnu Akademijas Vestis	L			12	10.00
Journal of the Academy of Sciences of the Latvian SSR					
Liteinoe Proizvodstvo	R			12	5.00
Foundry Production					
Metallovedenie i Termicheskaia Obrabotka Metallov	R	HB		12	7.00
Metallography and Thermal Working of Metals					
Metallurg	R	CB	$95.00	12	4.00
The Metallurgist					
Nauchnye Doklady Vysshei Shkoly	R				
Scientific Reports of Higher Schools					
Serila: Metallurgiia				4	6.00
Metallurgy					
Ogneupory	R			12	8.00
Refractories					
Pribory i Tekhnika Eksperimenta	R	ISA	$25.00	6	12.00
Instruments and Experimental Techniques					
Priroda	R			12	11.00
Nature					
Referativnyi Zhurnal	R				
Abstract Journal					
Serila: Mekhanika	R			12	18.00
Mechanics					
Serila: Metallurgiia	R	P	$20.00	12	67.00
Metallurgy					
Soobschenia Akademii Nauk Gruzhianskoi SSR	G			12	9.00
Reports of the Georgian Academy of Sciences	R				
Stal'	R	BISI	£20.12.0	12	10.00
Steel					
Stanki i Instrument	R	PERA	£4.4.0	12	8.00
Machine Tools and Cutting Tools					
Stekio i Keramika	R	CB	$80.00	12	6.00
Glass and Ceramics					
Svarochnoe Proizvodstvo	R	BWRA	£5.5.0	12	6.00
Welding Production					
Tsvetnye Metally	R			12	10.00
Nonferrous Metals					
Vestnik Akademii Nauk SSSR	R			12	13.00
Journal of the Academy of Sciences of the USSR					
Vestnik Kazakhskoi SSR	R			12	10.00
Journal of the Kazakh SSR					
Vestnik Akademii Nauk Ukr. RSR	U			12	6.00
Journal of the Ukrainian Academy of Sciences					
Zavodskaia Laboratoriia	R	ISA		12	13.50
Factory Laboratory					

PRESSURE-POURED STEEL

A key to lower-cost steel mill operations?

A technique, now being used to cast steel railway wheels, is undergoing pilot-plant operations designed to test its applicability to cast semi-rolled steel shapes.

by F. Weston Starratt

A controlled-pressure technique for pouring railway-car wheels, developed by Griffin Wheel Co., subsidiary of American Steel Foundries, may be the answer to the casting of semi-rolled steel shapes, thus bypassing soaking pit, and primary rolling operations. U.S. Steel Corp. feels that the idea has considerable merit and has reached an agreement with Griffin Wheel whereby experimental casting of semi-rolled shapes on a pilot-plant scale is taking place at its South Chicago works.

Development of the pressure-pouring technique dates back more than a decade to the time when Griffin was struggling to maintain its position in the competition between cast-iron, forged, and cast-steel railway wheels. Costs of cast-steel wheels had to be lowered, while at the same time a more uniform product made on a production-line scale was required. Thus, came about the idea of pressure pouring on a semi-automatic basis. Pilot-scale development began in 1950, and by 1953 commercial production was underway, first in Chicago and later at five Griffin plants in North America. Output is now on a production-line basis, with as few as three men operating one line. More than 1,000,000 wheels have been cast by the pressure pouring technique.

Casting car wheels

Before moving on to possible steel-mill applications, a look at the pressure-pouring technique as applied to foundry practice is in order. Griffin uses semi-killed steel containing 0.70 pct C and 0.70 pct Mn. Ten to 15-ton ladles are employed.

At a Griffin's pouring line, a ladle containing molten steel at about 2900°F is lowered into position, and, at the same time, the attached pouring tube drops into the molten steel. The chamber is sealed, and the only accesses are the vertical, refractory pouring tube, which extends to the bottom of the ladle, and connections with the pressure system. A permanent graphite mold is secured to the top of the chamber lid and connected to the pouring tube. At the turn of a switch, a predetermined pressure is automatically applied to the chamber. Steel rises in the pouring tube, flooding into the wheel mold. When the mold cavity has been filled, a refractory plunger shuts off the metal flow, and the mold is removed from above the chamber. The next mold is then secured to the chamber lid, and the process is repeated until the level of the steel in the ladle is within 3 in. of the bottom of the tube. Pouring of the 16-ton ladles used at Griffin's Kansas City plant requires 40 min.

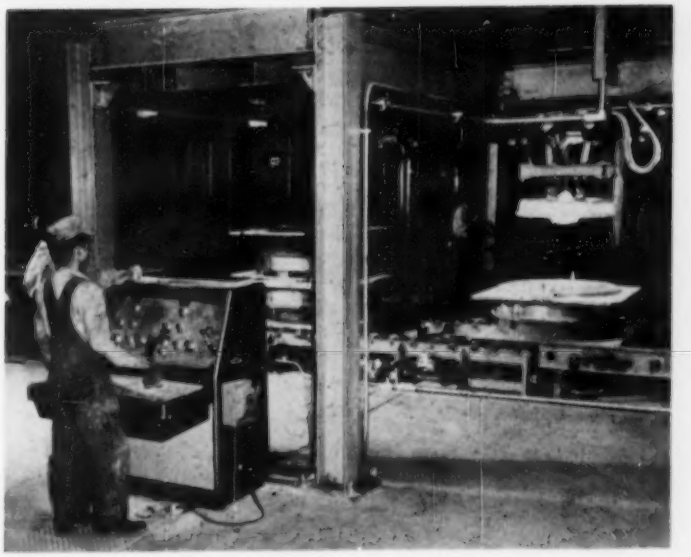
After a short cooling, the top of the mold is removed and the car wheel is grabbed by a conveying mechanism to be transported to machining and inspecting areas.

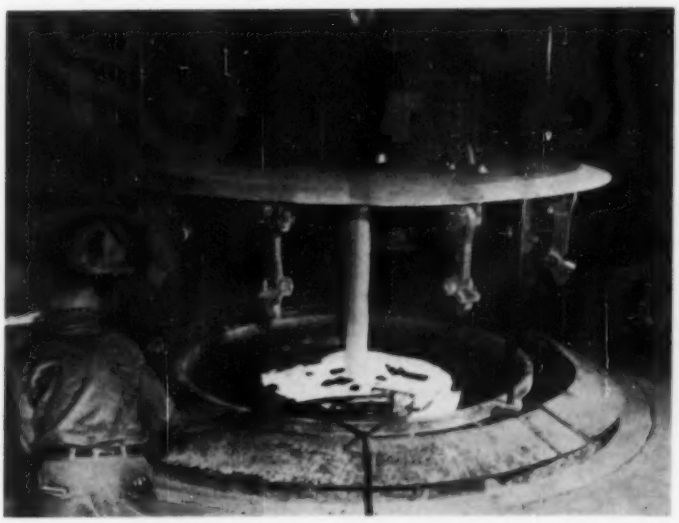
Not only is the pouring operation automatic, but cleaning of the castings has been practically eliminated, and only the bore of the wheel requires machining. With human judgment minimized and uniform casting pressures maintained, a much more standardized product is the result, and rejections have been reduced to a mere fraction of those of older techniques. Casting tolerances are extremely good, and easily meet the limit of 1/48 in. for a 33 in. diam wheel.

Besides Griffin, the only other firm utilizing the pressure-pouring technique is Lebanon Steel Foundry, to whom Griffin has granted a license. Lebanon is using the technique to produce a variety of castings, including turbine blades and valve bodies. Because of the variety of shapes cast, production is not on the same production-line basis as at Griffin.

Production of steel castings by pressure pouring, thus, has achieved commercial success. Perhaps the most difficult problem has been the life of the refractory pouring spouts required to handle molten steel up to 2900°F. After considerable research, pouring tubes have been developed with lives varying from three to thirteen heats.

Right, view of operations . . . wheel at left has just been poured. Crane at left removes the cope, while crane at right removes a previously-poured wheel.





Left, a close-up view of the pouring tank and cover.

Applied to steel mills

Application of the pressure-pouring process to the casting of semi-rolled shapes has been the subject of considerable investigation by Griffin's metallurgists, even prior to U.S. Steel's interest in the technique. The objective has been the elimination of high-cost primary rolling mills and soaking pits.

Many types of steel have been used in Griffin's experiments; they have varied in carbon content from 0.10 to 1 pct, and have included stainless types and boron steels. Perhaps the greatest interest has been in casting slabs, and Griffin has cast slabs measuring 5x37.5 in. x 15 ft from low-carbon steels. Stainless steel slabs also have been cast, and tube rounds up to 6 in. in diam have been cast without segregation. With reference to billets, experimental work has shown the possibility of casting 3x3 in. x 30 ft shapes from 150-ton heats in 40 min. Thus, pouring is rapid and stripping can also be accomplished with considerable speed.

These semis have been characterized by good surface finish, practically eliminating the necessity of scarfing and other surface treatment. One problem is that of center-line shrinkage; in practice it will probably necessitate a sealing of the ends of the cast shape in order to prevent oxidation stringers from forming. Because of this shrinkage, finished rolled shapes can not be cast. However, it appears likely that subsequent rolling can produce steel products of a quality equivalent to present commercial practice.

The difference between pressure pouring and present techniques appears to lie both in capital costs for steel-mill equipment and in production costs encountered in soaking pit, rolling, and scarfing operations. A further saving would result from increased yield of usable metal, resulting from an elimination of hot tops and cropped ends of semi-rolled shapes.

U.S. Steel's pilot-plant work in the next two years should answer the question of whether pressure pouring can be applied commercially to steelmaking.

What are the, COMBINED EFFECTS OF TEMPERATURE AND VERY HIGH PRESSURE ON METALS?

The inception of high-pressure, high-temperature operations has opened many new avenues of investigation. The author discusses the possible effects of both on electrical resistance, ductility, hardening, and other mechanical and structural properties of metals.

by R. B. Fischer

One of the greatest potential fields for metallurgical research was opened when means were developed to apply high temperatures to materials under very high pressures. The background for the subject was built over the years by the integrated efforts of many individuals, but the development of apparatus by General Electric Co. fired the imagination of reseachers.

To those whose routine efforts are devoted to producing and marketing a metal product, there will be an understandable reserve about getting excited over this subject, since the high-pressure, high-temperature cells available to date are small. On the other hand, the Engineering Supervision Co. recently offered to provide considerably larger cells than those used to date. No technical barriers seem to bar the construction of larger cells when needed; therefore, there is little cause for concern on this particular point.

More important is the question of what can be accomplished by the use of combined temperature-pressure operations. Here researchers, now adequately equipped, are striving for answers. Their efforts will bear close observation, because the results might bring about a drastic change in materials as we now know them. The use of high-temperature, high-pressure apparatus in studies of materials is so vast as to appear limitless. Facing this scope of possibilities, can one honestly maintain doubtful reservations about the potential of this subject? Advances are being made already in developing basic knowledge which will, at a minimum, feed back to help understand and improve present processes.

Effects on melting temperatures

For practical purposes, the melting temperature of a metal has long been treated as a constant. For example, temperature calibrations are based sometimes on the melting point of a pure metal. In the future, it is likely that the question, "What is the melting temperature of iron?", will be recognized by technologists as demanding the counter question, "At what pressure?". Recent work described by Strong[1] illustrates the point. Strong's work and others[2, 3, 4, 5], has been used in Fig. 1 to show the relationship of melting temperatures versus pressure for several metals.

At this time, the Clapeyron-type equation, $dT/dP = (\Delta V)/L$, is useful for predicting the effect of pressure on melting temperatures. The expression dT/dP is the change of melting temperature with a change of external pressure. If the proper units of T_m, the melting point at zero pressure, ΔV, the volume change on melting, and L, the latent heat of fusion, are substituted in the equation, fairly good estimates can be made. For those metals that expand on melting, an increase in pressure generally results in a higher melting temperature, since pressure tends to prevent expansion (and melting). This situation is overcome by increasing the temperature to the extent that melting can occur. In general, an opposite relationship exists for those metals that contract on melting; an increase in pressure lowers their melting temperature.

There is a question as to the extent that these relationships hold as higher pressures and temperatures, not yet attainable in present apparatus. It is known for bismuth, that the melting temperature decreases with increasing pressure to a point where the metal transforms into a new solid phase. This new phase has a melting temperature curve which then increases with pressure. The melting temperature curve for rubidium increases with pressure to a maximum and then decreases, but this is unusual, as Strong[1] has indicated. For other metals, melting temperatures have been found to increase or decrease as expected up to the limits of present apparatus. Will these relationships continue; will phase transformations occur; and what are the ultimate results of pressure application to metals? These are interesting questions for conjecture, but what can be done with the knowledge already at hand on the effects of pressure on melting temperature of metals?

R. B. FISCHER is chief, physical metallurgy div., Battelle Memorial Institute, Columbus, Ohio.

So far as is known, no industrial use has been made of the information that melting temperatures of metals may vary with pressure. The author's opinion is that, in time, uses will develop as the facts become better known and people dwell on possible applications.

Melting temperature information is of importance in gaining new information about the solid and liquid states of metals. It increases our scientific understanding of metals, and, in turn, this information should feed back to promote better industrial processes.

One scientific area may be cited in which melting temperature versus pressure data are important. In Strong's article[1], he shows how geophysical information of the Earth's structure is being advanced. Previously, data on the melting temperatures of iron were extrapolated to build theories on the core structure of the Earth. New high-pressure data have shown there was considerable error in the extrapolations. Because of the limits of present pressure apparatus, extrapolation is still necessary, but accuracy has been improved, and old formulae can be revised to match more realistic values. Much more work is needed, but significant progress has resulted from the recent high-pressure, high-temperature work.

Melting temperature data are valuable to researchers involved in high-pressure studies. Strong[1] has pointed out that thermocouples might be used in high-pressure apparatus at temperatures higher than the *melting points* of the thermocouple metals! As another example, a platinum heating element or structure in a high-pressure device might be retained in the solid state at 3700°F, although we usually think of platinum as a liquid at about 3225°F. Molten germanium at the relatively low temperature of 1000°F might be brought into contact with another material, if desired. Usually, a temperature of about 1650°F is considered necessary to melt germanium. Aluminum and germanium have the same melting temperature at about 325,000 psi. Thus, limitations of commonly expressed constants for melting point can be removed to some extent from research activity.

Another use of melting temperature data is in the design of new high-pressure cells. For example, liquid germanium could be confined under pressure, and, by cooling it below its freezing temperature, very high pressures could be produced. As Hall[2] has mentioned, the behavior of germanium is such that it is an excellent standard for very high-pressure calibration work.

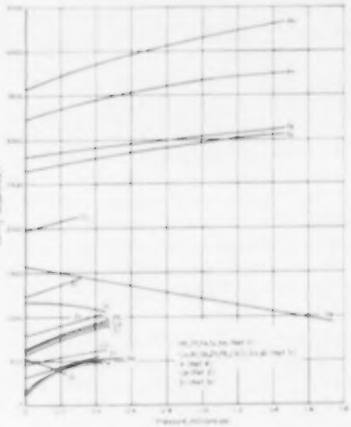


Fig. 1—Melting temperatures of several metals at various pressures.

Effects on phase changes and shifts

Several outstanding effects of pressure on phase changes of metals have been found. Butuzov[3] has presented the phase diagram of bismuth with pressure as a factor as shown in Fig. 2. Such data are useful in the calibration of high-pressure, high-temperature devices. Methods are being developed to determine the crystal structures of the bismuth phases. These phase changes are reversible; therefore, X-ray diffraction data must be obtained while the metal is under pressure. The uses of a diamond cell for such studies has possibilities.

Hall[3] has reported that a hitherto unknown form of tin has been produced at high temperature and high pressure. No doubt other new phases in metals will be found in the future.

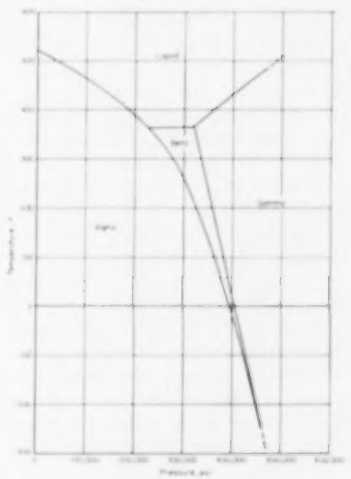


Fig. 2 Phase diagram of bismuth (Ref. 3).

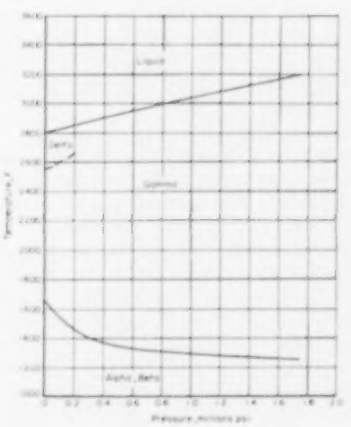


Fig. 3—Effect of pressure on phases of iron (Ref. 1).

Before high-pressure, high-temperature data were available, calculations were made on the transformation of α to γ iron under high pressure. The calculated values were used to help explain the history of meteorites. Laboratory data, such as that given by Strong[1], reveal that the theoretical values do not check with the measured values (Fig. 3). From this information, theory can be revised and a better understanding of meteorites obtained. Possibly, the information on meteorites will assist in solving re-entry missile problems.

The α to γ transformation temperature of iron is lowered by high pressure, as expected. (Alpha to gamma involves a contraction, which should be favored by pressure.) This effect will be studied intensely in the future because of the basic importance of this transformation to the steel industry. Efforts are now under way on such studies with the objective of developing steels with better properties. One good possibility is that the use of high pressure during heat treatment of steels should result in finer-grained steels with better properties. This is based on the principle that less grain growth should occur at the lower transformation temperatures possible with high pressure.

Hall[2] has mentioned a heat treatment of steel under high pressure. The steel is raised to the transformation temperature, and pressure is applied suddenly to *freeze* the new structure. Exceptional ductility and *through* hardening are claimed.

In addition to forcing materials into new phases (e.g., the reversible phases of bismuth) and the lowering of transformation temperatures, high pressure can be expected to cause shifts in composition from those limits now established on constitution diagrams. Thus, high pressure adds to the present two-dimension binary diagrams, but it certainly complicates the presentation of ternary and higher diagrams.

Phase shifts may offer opportunities, for example, in increasing the solubility of one metal in another. This could result in enhanced precipitation hardening of certain alloy systems and added corrosion resistance. Compositions of alloys may be shifted, resulting in an alloy that is less brittle than its counterpart of like composition made at low pressure.

High pressure may be a means of producing alloys containing a volatile constituent, an otherwise difficult and sometimes impossible accomplishment.

Effects on structural features

Casual observation with the aid of a microscope reveals that there are sizeable flaws in most materials. Such flaws may or may not be harmless. Voids or fissures, difficult to avoid in ordinary processing, might be closed or removed by use of high-pressure devices.

A tendency toward disorder of atom arrangements is produced in general when the temperature of a metal is raised. Disorder to some extent can be considered a natural condition in metals. There is an effort to make specimens of a more orderly structure; tiny metal *whiskers* are examples. These are in the form of very fine fibers and are considered to contain few dislocations. High pressure may be a means of providing an ordered structure in a body of large dimensions, in that pressure might assist in moving atoms toward ordered positions. It is possible that elevated temperatures to provide atom mobility would be necessary. One approach would be to allow metals to solidify very slowly under high pressure. A thin section of a metal with a perfectly ordered structure might have high-strength properties, no ductility at room temperature, and unusual toughness characteristics.

The conventional preparation of non-ductile metal compounds, such as tungsten carbide, must result in far-from-perfect crystals. Flaws are apt to result from the cooling stresses set up in the non-ductile crystals. The values obtained from property measurements on such imperfect crystals are likely to be lower than those obtained on sound crystals. High-pressure apparatus might be used to prepare sound crystals of compounds. For example, the crystals could be subjected to high pressure, heated intensely, cooled, and then returned to atmospheric pressure.

Complete removal of voids from bodies made from metal powders is most difficult. The use of high pressure should nearly eliminate voids. There is the possibility of encapsulating powders in an evacuated container and subjecting the assembly to very high pressure and the necessary temperature for maximum densification.

For a considerable time, the effect of pressure on semiconductors has been of interest. Lattice spacing has an influence on semiconductor energy bands. Long[6] noted that the density and mobility of electrons may decrease with pressure. The resistivity and Hall coefficient may rise with increasing pressure. For one p-type silicon crystal, Ryabinin[7], et al., reported that the electrical resistance was reduced with increasing pressure.

Vereshchagin and Zubova[8] noted that pressure-induced viscous flow of materials, including metals, is affected only by the number of external electrons present in the atom. They reported that the critical shear stress, which is inversely proportional to compressibility, is independent of type of crystal lattice and total number of electrons per atom.

Reaction rates and diffusion

New studies can be made on the reactions of metals with other substances under high pressure. It will be interesting to learn of the extent to which such reactions can be prevented or forced to proceed or reverse. The relative volumes of the reactants and products are expected to permit an estimate of the influence of high pressure on such reactions.

Diffusion rates are very likely to be influenced by high pressure. In the case of self-diffusion, there are indications that pressure has a retarding effect. This seems reasonable because pressure should crowd the structure and thus make self-diffusion more difficult.

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COLUMBIUM METALLURGY . . .

A report on the Lake George meeting.

by B. S. Shabel of the Knolls Atomic Power Laboratory of General Electric Co.

The first session of the AIME Columbium symposium was devoted to various aspects of the fabrication of columbium and its alloys. The opening paper was presented by P. J. Gripshover (Battelle) who described a pressure bonding technique for the production of solid-phase self bonds in plates, rods, and complex flat-plate sub-assemblies. Bonding was achieved by heating at 1150°C for 3 hr under an inert gas (He) pressure of 10,000 psi. The process has been extended to the production of columbium-clad UO_2 and cermet fuel elements and has even permitted tubes to be formed directly from columbium sheet. This technique may also be extended to columbium alloys.

R. T. Torgerson (Boeing) then presented an evaluation of the forming characteristics of Cb-10 Ti-10 Mo, Cb-33 Ta-0.75 Zr, Cb-10 Ti, and Cb-0.75 Zr alloys. From consideration of strength, ductility, and sub-zero transition temperature, the best alloy appeared to be the interstitially-hardened Cb-33 Ta-0.75 Zr material.

A. I. Lewis (Westinghouse) outlined the results of a study by Begley and Lewis on the effect of carbon on the workability and mechanical properties of columbium. It was observed that, up to 0.072 weight pct C, the Cb-C alloys retained workability, but that, at higher carbon contents, workability decreased. A 0.75 weight pct C alloy was tested and showed cracking after 5 pct cold rolling, whereas a 1 pct C alloy was cold rolled 15 pct before cracking occurred. The loss of workability was related to the presence of a brittle Cb_2C phase in the grain boundaries. However, it was also observed that carbon content did not affect the yield strength in the temperature range −196° to 200°C.

A practical technique for increasing the cold workability of columbium alloys was described by J. De Mastry (Battelle). This consists of forging the cast ingot to approx a 40 pct reduction in air at 540°C, and a subsequent rolling at 540°C to sheet about 0.100 in. thick. Alloys of columbium and chromium, zirconium, and molybdenum and a ternary Cb-Ti-Mo alloy could, after successful break-up of the as-cast structure, be cold-rolled to 0.2 mil foil without difficulty.

The effect of gaseous contaminants was next discussed by H. E. McCoy (Oak Ridge). Nitrogen in excess of 1000 ppm, hydrogen in excess of 500 ppm, and oxygen in excess of 3000 ppm were observed to cause serious embrittlement. Nitrogen and oxygen, at elevated temperatures (1010°C) did not seriously affect ductility, and, in fact, increased the strength of the metal. Creep tests at 982°C, however, showed that the presence of hydrogen or water vapor increased the creep rate.

The possibilities of improved oxidation resistance through the use of aluminum-dip coatings were pointed out by R. G. Carlson (G. E.). A 15 pct Si-Al dip coating provided up to 10 hr protection at 1260° and 1370°C for the commercial alloys F-48, F-50, and Fansteel 82. The optimum coating treatment required a 3 min dip at 925°C, followed by a 1-hr vacuum anneal at 1040°C. For longer periods of oxidation resistance, a longer time and/or higher temperature dip appeared necessary.

R. J. Van Thyne (ARF) suggested a means of reducing the oxygen content of columbium powder by utilizing the carbon-oxygen reaction. It was found that, by adding carbon up to the stoichiometric amount required to form CO and sintering in vacuum for 1 hr at 2000°C, the oxygen levels would be lowered without seriously affecting the residual carbon content of the sintered compact.

The final paper of the session was presented by S. J. Noesen (G. E.) who described the procedures evolved for the production of the F-48 and F-50 alloys by vacuum arc melting.

Mechanical Properties

The first afternoon session began with S. T. Wlodek (Union Carbide Metals Co.) presenting a summary of the oxidation behavior and mechanical properties of Cb-Ti-W alloys. The maximum oxidation resistance, up to 1400°C, was observed for compositions in the range of 5 to 15 weight pct Ti and in excess of 15 weight pct W. Oxidation resistance was associated with the formation of a Cb_2O_5-TiO_2-WO_3 type of columbate. Better spalling resistance was obtained by modifying the ternary alloy with molybdenum or vanadium; a representative quaternary alloy being Cb-10 Ti-20 W-3V. Molybdenum modifications were observed to give greater strength at elevated temperatures than vanadium modifications, although neither modification was as strong as a Cb-7 Ti-28 W alloy.

D. J. Maykuth (Battelle) reviewed the results of an investigation of columbium-base alloys for use in pressurized-water reactors. The binary alloys, Cb-7.6 atomic pct V, Cb-12.6 atomic pct V, and Cb-7.2 atomic pct Mo, showed superior strengths to Zircaloy 2 at temperatures above 400°C and much improved creep resistance at temperatures through 650°C. In this temperature range, ternary alloys of Cb-V-Mo and Cb-Ti-Mo also exhibited good strength and creep resistance.

The strain aging effect due to hydrogen in columbium was analyzed by B. A. Wilcox (Wright-Patterson AFB). Measurement of the temperature dependence of the rate of yield point return indicated an activation energy of 10,500 cal per mole, in rough agreement with that observed for the diffusion of hydrogen in columbium (9370 cal per mole). Similar activation ener-

gies were observed by dynamic modulus experiments, supporting the interpretation of hydrogen as the cause of strain aging.

Processing and the properties of F-48 alloy sheet were described by T. K. Redden (G.E.). Hot rolling above 700°C was required for initial rolling operations after extrusion of the ingot. Canning in molybdenum was required for this stage, after which the desired gage thickness could be reached by cold rolling. However, warm rolling in the range 200° to 300°C has proven best for good surface finish. The 0.2 pct yield strength varied from about 105,000 psi at 25°C to approx 65,000 psi at 1000°C, the elongation at this temperature being about 7 to 8 pct, while the stress for 1-hr rupture at 1371°C was 20,000 psi.

D. P. Laverty (Thompson Ramo Wooldridge) next presented the results of measurements of Young's Modulus. The modulus was observed to decrease linearly from 16.2 million psi at room temperature to 14.1 million psi at 820°C.

D. P. Gregory (CANEL—Pratt and Whitney) described a dislocation mechanism accounting for the creep and recovery behavior of columbium and a Cb-1 pct Zr alloy. The postulated mechanism involved the annihilation of unlike screw dislocations by cross-slip, the remaining edge dislocations accounting for the observed mechanical behavior. The creep behavior of columbium was considered to be governed by a forest intersection process below 930°C; the addition of 1 pct Zr retarded polygonization and retained the intersection process as rate governing up to 1130°C.

E. S. Tankins (Naval Air Material Center) presented the final paper of this session, dealing with the effect of strain rate, grain size, and temperature on the yield strength of columbium. A linear relationship between log stress and log strain rate was found, and it was indicated that strain rate was independent of grain size. A strain aging phenomenon was observed, and measurements of the temperature dependence of the rate of yield point return gave a value of 27 K cal per mole, suggesting that oxygen might be responsible for the aging. The yield strength was found to vary linearly with grain diameter raised to the $-\frac{1}{2}$ power. Tests at liquid air temperatures showed the absence of a lower yield point, higher yield strengths, and the presence of twins in the microstructure.

General Metallurgy

The General Metallurgy session on Friday considered several aspects of columbium metallurgy and covered a range of subjects from thermodynamic measurements to the mechanical properties of binary alloys.

The opening paper was presented by R. P. Elliott (ARF) who presented a probable Cb-N phase diagram up to the stoichiometric CbN composition. The solubility of N in columbium was found to be 0.25 weight pct at 1200°C and 2.5 weight pct at 2400°C. The X-ray data indicated the presence of the following phases: hexagonal close packed Cb_2N, tetragonal CbN (Cb_4N_4), fcc CbN, and hexagonal CbN.

The application of formal alloy theory to a wide range of columbium binary alloys was next described by A. E. Dwight (ANL). A definite sequence of crystallographic phases was observed with increasing alloy content, running from bcc → Cr_2O → σ → Laves → α Mn → hexagonal close packed variant → fcc variant. While no alloy system contained all the phases, the order of occurrence was always followed, e.g. Cb—Os, which shows the bcc → Cr_2O → σ — α Mn → ϵ phase (hexagonal close packed). Consideration was also given to the Cb-V-Zr and Cb-Ti-Mo systems.

G. H. Rowe (CANEL—Pratt and Whitney) presented work describing the recovery and recrystallization of a Cb-1 pct Zr alloy and the subsequent mechanical behavior. One hour at 1430°C was required for recrystallization of the 40-80 pct cold reduced material, whereas 1540°C was necessary to recrystallize the 20 pct reduced material. Recovery of hardness was observed at much lower temperatures and was related to the dislocation mechanism outlined by Gregory earlier in the conference. Short-time tensile tests at 980°C revealed a peak yield strength in the material reduced 20 pct, although the ultimate tensile strength increased with increasing cold work.

Recrystallization was also discussed by B. S. Shabel (KAPL) who spoke on the behavior of columbium-vanadium alloys. The activation energy (50 pct reduction) was found to be : Cb-0.6 atomic pct V and 1.6 atomic pct V-92 K cal per mole; and Cb-8.9 atomic pct V-118 K cal per mole. One hour at 1150°C recrystallized the 0.6 and 8.9 atomic pct V alloys while 1100°C sufficed for the 1.6 atomic pct V alloy; however, a possible grain-size effect was indicated here. Recovery effects were measured by a Fourier line-broadening analysis, and the textures of cold-rolled and recrystallized sheet determined. The cold-rolled texture was found to be basically (100) <011> and (112) <110>. The recrystallized texture of the Cb-0.6 atomic pct —V was (100) <011>, (111) <112>, whereas Cb-3.5 and 8.9 atomic pct —V alloys retained the (112) <110> component after recrystallization.

R. T. Begley (Westinghouse) presented work based on the flow and fracture characteristics of electron-beam-melted columbium. The observed work-hardening, ductile-brittle transition, and strength-temperature behavior were all consistent with the nature of a bcc metal.

The role of oxygen in the strain aging of columbium tested in tension and fatigue was described by J. Enrietto (U. of Illinois). Maximum strengthening was observed to occur at 500°C in tension ($\dot{\epsilon}$ = 0.04 per min) and at 400°C in fatigue (frequency = 3450 cpm). The strengthening effect of oxygen increased up to an oxygen concentration of 0.02 weight pct at these temperatures, but increasing concentration contributed little additional strengthening.

O. M. Katz (Westinghouse) presented data and calculations of the thermodynamic functions in the Cb-H system for temperatures from 225° to 513°C at hydrogen pressures from 0.01-760 mm of Hg. The relative partial-molar free energy of hydrogen in the Cb-H alloys increased with increasing composition at a given temperature and increasing temperature at a given composition. The relative partial-molar enthalpy of hydrogen decreased from —7.59 k cal per mole of hydrogen at a mole fraction of 0.035 to —10.40 k cal per mole at a mole fraction of 0.405 with an inversion point at a mole fraction of 0.350. Relative partial-molar entropies decreased from —5.54 k cal per mole to —15.78 k cal per mole of hydrogen. It was indicated that the hydrogen bond strength in the lattice increased up to a mole fraction of 0.350, increasing hydrogen tending towards saturation and a marked reduction in bond strength.

The final paper of the morning by Braun, et al (Metallwerk Plansee) was presented by R. I. Jaffee (Battelle) and considered the properties, particularly workability, of various Cb-W and Cb-Mo alloys. In both alloy systems, a range of compositions in the middle of the phase diagram were unworkable by rolling or die forging below 1000°C, although high-columbium alloys (<20 atomic pct Mo, W) could be cold rolled and high tungsten or molybdenum alloys could be rolled or swaged after heating to 1000°-1600°C in a hydrogen atmosphere.

Oxidation and Corrosion

S. T. Wlodek (Union Carbide Metals Co.) began the session with a discussion of the Cb-Al-V system. This alloy system appeared promising from the viewpoint of oxidation resistance and good mechanical properties for temperatures below 1200°C.

H. A. Fisch (KAPL) presented the results of an investigation of the steam corrosion behavior of Cb-V

alloys. The corrosion rates in 1500 psig steam at temperatures from 360° to 482°C decreased with increasing vanadium content. The oxide composition varied from Cb_2O_5 for the least protective film to CbO_2 for the most protective film.

The ignition phenomenon was next discussed by J. W. Clark (G.E.). Columbium was reported to ignite at 1470 ± 10°C, if air mass flow rates exceeded 2000 lb per sq ft per hr. Alloy additions of molybdenum, titanium, and tungsten decreased the ignition temperature. However, a short preoxidizing treatment at temperatures below the ignition range increased the ignition temperature and/or the maximum heat flux which could be supported without ignition.

H. Inouye (Oak Ridge) presented the results of an investigation of the oxidation of columbium at low oxygen pressures. Oxidation occurred by stages, consisting of; solution of oxygen, internal oxidation, film formation, and an oxide-gas reaction. Internal oxidation commenced with the precipitation of CbO at and below the surface. The third stage was characterized by the formation of CbO_2 while the final stage resulted from the reaction between CbO_2 and oxygen to form non-protective Cb_2O_5.

R. E. Pawel (Oak Ridge) reported the results of further investigations of the study of oxide-film formation. He found that specimens of (100) orientation showed a relatively long period of protective oxidation with a slow transition into the non-protective region, but that specimens of (111) or (110) orientation oxidized protectively for short periods and underwent an abrupt transition to the linear stage of oxidation.

The hot-water corrosion behavior of Columbium and Columbium alloys was next described by W. D. Klopp (Battelle). Improved corrosion behavior was achieved by alloying with V, Ti, Zr, and Ti-Cr additions, with no difficulties resulting from oxygen or hydrogen absorption during corrosion. The Cb-V and Cb-Zr alloys were considered to be the most promising for applications in thermal reactors.

The final paper of the conference was presented by J. Ong (Washington University). Prof. Ong reported a mathematical correlation of the form, rate $= A' \left(\frac{-\Delta H}{RT}\right) \theta' = K_R \theta'$, where K_R is the reaction rate constant and θ' is the fraction of the interstitial sites on which absorption of oxygen had occurred. The mathematical expression for describing the effects of temperature and pressure on the oxidation rate was based on the assumption of an interstitial solid-solution being formed by oxygen in columbium. While calculated points were not in precise agreement with the available experimental data, the trends of the theoretical curves were borne out by the data. The correlation was also applied, with promising agreement, to Cb-Ta alloys.

The proceedings of the conference will be available by early 1961.

RESPONSE OF METALS TO HIGH VELOCITY DEFORMATION . . .

A report on the Estes Park conference.

by J. B. Clark of The Dow Metal Products Co.

A few years ago, C. S. Smith[1, 2] pointed out that the passage of high-pressure shock fronts through metals produced unique structural and property changes which should be of interest to metallurgists. Although considerable work has been carried on in this field in the last few years, much of it remains unpublished or has been reported outside of the general metallurgical literature.

On July 11th and 12th at Estes Park, Colo., a technical conference was held for the purpose of reporting and discussing recent work. A complete review of all the papers is not possible here; this report attempts to summarize only those papers dealing with the metallurgical effects produced by the passage of high-pressure shock waves through metals and alloys.

As a general theoretical background for the conference, J. Weertman discussed the unique properties of high-velocity dislocations. Eshelby's analysis[3, 4] for a moving-edge dislocation was developed a little further. It was shown that contrary to the usual situation, edge dislocations of like sign attract each other (unlike repel) when the dislocations are traveling above the Rayleigh wave velocity. Screw dislocations do not change behavior at high velocities. Thus, at high velocities it may be possible for a group of edge dislocations to coalesce and form a microcrack. Weertman pointed out that the edge-type dislocation described by Eshelby[5] can move at supersonic speeds, and this type of dislocation may be necessary for Smith's dislocation interface[1, 2], between shocked and unshocked regions.

G. E. Duval reviewed the general properties of shock waves. He showed that by means of explosive loading techniques, a plane compression shock wave can be transmitted through a solid at speeds near 5 km per sec. Shock fronts may involve pressures as high as several hundred kilobars [1 kilobar (kb) equals 14,504 psi = 986.9 atm] and may compress the metal as much as 20 pct. The Rankine-Hugoniot equation, which relates the internal energy of the shocked state to the

initial and final pressures and volumes of the material, was derived. (For a published development of this relationship, see Rice et al[6].) When combined with an equation of state, this relationship yields the Hugoniot curve, which is the locus of all P, V states of a material attainable by propagating a shock wave into a fixed initial state. On a P-V diagram, the Hugoniot curve is curved concave upward to the left. The slope of this curve will decrease somewhat at the Hugoniot elastic limit (E_I) where plastic flow begins and also at a higher pressure (designated the transition pressure P_I), if a high-pressure phase exists in the P-V system. If the material undergoes a phase change at higher pressures, a second plastic wave (P_{II}) characteristic of this phase will follow the first plastic wave. Conversely, the existence of a double wave has been taken as evidence of a polymorphic phase change.[7]

The response of metal to shock waves was discussed from several aspects:

A) The Dynamic or Hugoniot Elastic Limit

The physical process occurring at this point on the Hugoniot curve is not completely known. F. S. Minshall noted that the Hugoniot elastic limit is larger than the quasi-static yield strength even when recalculated as a one-dimensional yield strength. The pressure of the elastic wave varies widely from 2 to 25 kb, depending on the alloy and the shock conditions. E. G. Zukas and C. M. Fowler showed that in iron, rolling prior to shocking, increased the pressure and velocity of the elastic wave. Further study is needed.

B) Plastic Wave Interaction at an Interface

A plastic shock wave normal to an interface will, under appropriate conditions, reflect as a rarefaction wave. Smith[1] has shown that in a a metal with two plastic waves, interaction of the reflection of the first plastic wave with the slower second wave leaves a visible interaction zone of lower hardness near the surface. Fowler, Minshall, and Zukas have developed a method of determining the Hugoniot and transition pressure, P_I, by measurement of the interaction zone. This method permits economical screening of alloys and rapid approximation of the transition pressures without elaborate equipment.

Smith reported on the interaction of shock waves traveling parallel to a common interface in a composite sample of iron and copper rods. The system centers itself upon the plane of interface so that the shock in both materials moves with velocity corresponding to the average density and compressibility.

C) Shock Induced Deformation Markings

Zukas and Fowler found that in annealed iron, Neumann twin markings, formed by shock loading, could be suppressed by prestraining before shock loading. The twins survived high-pressure phase transitions without change in shape. The evidence presented seemed to indicate that the twins were caused by the compressive elastic wave. It is difficult to see how an elastic wave could produce plastic deformation—the formation of twins. Smith suggests that the pressure-distance profile of the elastic and plastic waves moving through the material will show another discontinuity between E_I and P_I which generates these twins.

Shock-induced twins can form in very small ferrite areas. Zukas observed twins in the fine ferrite of the pearlite of a 1.0%C steel. H. P. Tardif, F. Claisse, and P. Chollet showed that shock-induced twins in proeutectoid ferrite cross into the pearlite grains indicating an orientation relationship between proeutectoid ferrite and pearlitic ferrite. They suggest that shock-induced twinning may be used to study grain orientation, sub-boundaries, and the continuity of phases.

The role of crystallographic orientation of the metal to the shock direction is still obscure. Zukas and Fowler noted that more twins were produced in iron when shock was perpendicular to the rolling direction. Smith shocked four copper single crystals parallel to [110], [111], [100], and [112] directions. All crystals except the [100] crystal showed twins. The [100] crystal showed no markings although this crystal suffered the greatest change in dimensions from the shock loading. Single crystal studies are continuing.

D) Change of Strength Properties Due to Shock

G. E. Dieter showed that a given amount of strain produced by compressive shock loading increases the hardness of columbium, iron, and nickel to a much greater extent than the same amount of strain by rolling. Holtzman and Cowan illustrated this point in austenitic manganese steel in which a 422 kb shock rasied the yield strength from 58,000 to 174,000 psi but only reduced the elongation from 60 pct to 19 pct. Microstructure studies reveal no obvious strengthening process. Presumably a critical arrangement of point defects is formed by the shock, but the exact strengthening mechanism is unknown.

E) Phase Transformations

Zukas, Fowler, and Minshall have reinvestigated the 130-kb phase transition in iron reported by Bancroft et al[7]. The transformation does not destroy pre-existing twins or grain boundaries, and the pole figures remain unchanged. Cold working of the iron raised the pressure of E_I and P_I about 6 kb. The transition pressure (P_I) for iron-nickel and iron-chromium alloys has been studied. The presence of some α phase is necessary for the high-pressure phase change. Although it has been determined that this is not an α-γ phase change, the real nature of the transition is still unknown.

Holtzman and Cowan compared the transformation markings in austenitic manganese steel as produced by cold rolling and shock loading. After 70 kb, the microstructure is very similar to the coarse structure of cold-rolled austenitic steel. At 422 kb, the markings become relatively discrete and uniformly distributed.

In the discussion, it was pointed out that the high pressure phase must be more dense and probably forms rapidly by a shear process. Also, care should be taken in attempting to predict phase changes during pressure-volume changes from the equilibrium phase (T-x) diagram of the alloy.

Status of the Field

The status of the field was reviewed at the conclusion of the conference. Two general areas require further work. First, the physical structure of the shock front requires study: its thickness, dislocation array, and mechanism of movement through the lattice need to be determined. Secondly, accurate studies of the pressure profile of the shock front are necessary. The nature and broadness of the Hugoniot elastic limit, the possible existence of a low-pressure plastic wave for production of Neumann twin bands and the rise time of P_I and P_{II} should be studied. One may anticipate further discoveries in this relatively new field of metallurgical research.

Publication of the Proceedings of this technical conference is planned within nine months. These Proceedings, which will include the pertinent discussion, will constitute a worthwhile addition to the literature.

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[3] J. D. Eshelby: Uniformly Moving Dislocations, *Proc. Phy. Soc.*, vol. 62A, 1949, pp. 307-314.

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[6] M. H. Rice, R. G. McQueen, and J. M. Walsh: Compression of Solids by Strong Shock Waves, *Solid State Physics*, vol. 6, Seitz and Turnbull (ed): *Acad. Press, Inc.*, New York, 1958.

[7] D. Bancroft, E. L. Peterson, and F. S. Minshall: Polymorphism of Iron at High Pressures, *J. Applied Physics*, vol. 27, 1956, pp. 291-298.

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NEWS OF YOUR PROFESSION

Oct. 5-7 Dates for Rocky Mountain Minerals Conference

Oct. 5-7 are the dates of the Sixth AIME Rocky Mountain Minerals Conference, scheduled for the Newhouse Hotel in Salt Lake City.

Sessions of particular interest to metallurgists will be held on Thursday, Oct. 6 with papers on *Reagent Systems Design* by J. D. Vincent, American Smelting & Refining Co.; *Electrolytic Tungsten and Molybdenum* by D. H. Baker, Jr., and J. B. Zadra of the Reno Metallurgy Research Center of the US Bureau of Mines; *Role of Micro—organisms in Leaching Processes* by a staff member of Kennecott Research Center; *The Vernal Phosphate Project of San Francisco Chemical Co.*, by D. L. King, San Francisco Chemical Co.; *The Silverton Project of Standard Metals Corp.*, by Edgar T. Hunter of Standard Metals; and *Mining The Sea Floor* by L. E. Shaffer of the University of California.

Social events will include a Welcoming Luncheon on Wednesday, Oct. 5 in the Newhouse Hotel and a mining companies' and suppliers' cocktail party later during the day. On Thursday there will be a Minerals Luncheon with guest speaker, Joseph L. Gillson. For Friday afternoon there is scheduled a trip through one of the west's most modern plants for research, development, and production of solid-fuel propellents and rocket engines, the Magna, Utah. plant of Hercules Powder Co. That evening an informal dinner-dance is scheduled for the Newhouse Hotel.

Requests for hotel reservations should be sent to Utah Section, AIME, 2260 Wilson Ave., Salt Lake City 8, Utah.

Papers on Imperfections

Papers are being sought for a technical session on *Direct Observations of Imperfections in Crystals*," which will be conducted at the Annual Meeting of The Metallurgical Society of AIME. The meeting will be in St. Louis in February, 1961.

The papers should be concerned with unpublished research. Presentation time will be 10 min, and 5 min will be allowed for discussion.

Abstracts of 500 words or less should be submitted *before* October 1, for consideration. Authors should state, when submitting abstracts, whether a written version of their paper can be submitted before February 1, 1960 for possible publication in a unified form. Abstracts of 500 words or less should be sent to:

Dr. J. B. Newkirk
Research Laboratory
General Electric Co.
P. O. Box 1088
Schenectady, New York

Education Council Officers Proposed

The Nominating Committee of the AIME Council of Education has proposed A. W. Schlechten as vice chairman for one year and chairman-elect, according to J. D. Forrester, chairman of the council. The current vice chairman, Charles V. Kirkpatrick, automatically becomes chairman.

Other nominations were R. L. Whiting, secretary-treasurer for one year, and J. J. Schanz, Jr., executive committee member at large for three years.

All officers and executive committee members take office in Feb. 1961. The Council of Education's bylaws provide that alternate slates may be proposed in writing to the Chairman of the Council of Education by ten or more members prior to Dec. 1.

NOTICE TO AUTHORS

Make sure that manuscripts are typed double-spaced before submitting them to the JOURNAL OF METALS. Failure to do so results in retyping, chance of error, and needless delay. To facilitate matters, submit papers in duplicate and enclose illustrative material such as drawings and glossy photos at the same time. Such illustrations should be suitable for reproduction.

Set Agglomeration Symposium For April In Philadelphia

Tentative programing has been completed for the International Symposium on Agglomeration, scheduled for the Sheraton Hotel in Philadelphia April 12-14, 1961. The Symposium is under the joint sponsorship of The Metallurgical Society and the Society of Mining Engineers, AIME.

Since the subject is one of natural interest for iron and steel men, the Symposium has been purposely planned to follow the 1961 Conference of the National Open Hearth Steel Committee and the Blast Furnace, Coke Oven, and Raw Materials Committee, meeting at this same Sheraton Hotel April 10-12.

Emphasis will be on the fundamental aspects of agglomeration; both ferrous and nonferrous ores, as well as carbonaceous materials, ceramics, and other nonmetallics will be under discussion. Sintering, pelletizing, nodulizing, briquetting, powder metallurgy, ceramic bonding, and related processes will be included.

The tentative session alignment is as follows: Wednesday morning, April 12th, Characterization of Raw Materials; Wednesday afternoon, April 12th, Preparation of Raw Materials; Thursday, April 13th, a whole-day discussion of The Agglomeration Process; Friday morning, April 14th, Evaluation and Utilization of Products; and Friday afternoon, April 14th, Economics and Process Design.

Already, 18 papers have been deemed acceptable for presentation, with another 18 potential papers. The papers will be presented in abstract form and then discussed. All papers accepted for the Symposium will be preprinted and mailed to pre-registrants well in advance in order that appropriate discussions can be prepared. The abstracts will be in English, French, and German.

The keynote address for the Symposium will be sounded by Ronald R. McNaughton, AIME President for

(Continued on page 708)

1961. He will address the luncheon on Wednesday, April 12th.

A program for the ladies in attendance has been arranged, and they have been promised a free luncheon at the Hercules Country Club during their all-day tour of the Longwood Gardens and the du Pont Museum.

The Symposium Proceedings, together with the technical papers and discussions, will be published promptly after the Symposium and mailed to all registrants.

Registration fee for AIME members is tentatively set at $25; $27.50 for nonmembers. The fee entitles the registrant to one set of preprints and one volume of the Proceedings. Student registration, $5; no preprints or Proceedings volume is included.

For further information, write W. B. Stephenson, General Chairman, AIME International Symposium on Agglomeration, P. O. Box 635, Paoli, Pa.

AISE Convention

Developments in Soviet ferrous metallurgy will be the subject of one of the 45 tecnhical papers to be presented at the 1960 Iron and Steel Convention and Exposition which will be held in Cleveland Public Auditorium, September 27-30.

The Soviet report, *Present Achievements and Immediate Prospects of Development in Ferrous Metallurgy*, predicts the production of 91 million tons of steel by 1965 and more than 120 million tons by 1970. It will be presented by Ivan Denisenko, economist, Economic Research Institute, and Roman Belan, metallurgical engineer and head of Iron and Steel Industry Dept., State Planning Commission, USSR. The paper will indicate new Soviet facilities placed in production this year, including five new blast furnaces, ten large-capacity open-hearth furnaces, three electric furnaces, six big rolling mills, a sheet steel cold-rolling department, steel and iron pipe production facilities, and eight coking batteries.

Among the more significant papers will be a report on the Open-Coil Annealing Process, a technique which is said to reduce the annealing cycle to 10 hr. Details will be made known on production aspects of the Dwight-Lloyd McWane Ironmaking process, a new direct-reduction process for foundry applications. And a fourth oxygen steelmaking process will be introduced—a British oxygen-lance process in a specially designed open-hearth furnace, called the Ajax furnace.

Symposium on Metallurgy in Army Will Be Featured at Fall Meeting

Start planning now for the Fall Meeting of The Metallurgical Society in Philadelphia Oct. 17-20. The registration desk, located in parlors A and B, will be open on Sunday from 5:00 to 9:00 pm; Monday through Wednesday from 8:00 am to 4:00 pm; and on Thursday from 8:00 am to 3:00 pm. Registration fee for AIME members is $5. For nonmembers the fee is $10, and $1 for nonmember students. There is no fee for AIME student members.

A symposium on *Metallurgy in the Army—Requirements, Applications, and Research*, sponsored by the Institute of Metals Division, will be one of the features of the meeting. The symposium is the second in a series of armed services presentations intended to acquaint those engaged in non-military research and development activities with the scope and extent of corresponding military activities.

Preceding the symposium there will be a luncheon at which John R. Townsend of the Office of the Department of Defense for Research and Engineering will speak on *The Role of Metals in National Defense*. Tickets for the luncheon will be available at the Registration Desk.

The Institute of Metals Lecture, formerly known as the IMD Annual Lecture, and customarily presented at the Annual Meeting, will be given at the Fall Meeting this year. Entitled *The Work Hardening of Metal Crystals*, the lecture will be given by Prof. N. F. Mott, F. R. S., of Cavendish Laboratory in the Department of Physics, Cambridge University, Cambridge, England, at 11 a.m. on Tuesday, Oct. 18, in the West Ballroom.

The annual Fall Dinner of the Society will be held in the Pennsylvania Room on Tuesday, Oct. 18, preceded by a cocktail party in the Constitution Room for all persons holding dinner tickets. Dress is informal (business suits).

The traditional Powder Metallurgy luncheon is scheduled for Thursday, Oct. 20, between morning and afternoon technical sessions.

Tickets for the Fall Dinner and all luncheons will be on sale at the Registration Desk. In order to assure adequate facilities, it is suggested that all tickets be purchased as early as possible. Tickets for the Army Day Luncheon *must* be purchased *before* 9:30 a.m. on Monday.

Technical sessions: The program will include sessions sponsored by the three divisions of The Metallurgical Society.

In addition to the symposium, *Metallurgy in the Army*, the Institute of Metals Division through its Corrosion Resistant Metals Committee, will sponsor a symposium on *Recent Advances in the Metallurgy of Stainless Steel*. A symposium on *Ingot Structure Control* will be arranged and sponsored by the Melting and Casting Committee, Institute of Metals Division. *Diffusion* will be the subject of a symposium conducted by IMD's Chemistry and Physics of Metals Committee. The same committee will sponsor a symposium on Tuesday on *Effect of Extremely High Pressures*.

IMD's Titanium Committee will sponsor a session on Tuesday on *Titanium Versus Steel In Pressure Vessels for Hydrospace, Aerospace and Space*. A symposium on *Mechanical and Metallurgical Principles Affecting Hot Extrusion* will be sponsored by the Non-Ferrous Metallurgy Committee.

A two-part *Symposium on Metallic Moderators and Cladding Materials* will be presented on Wednesday by the Nuclear Metallurgy Committee. Another two-part symposium will be *The Decomposition of Austenite by Diffusional Processes* which the Ferrous Metallurgy Committee is sponsoring.

The Structural Materials Committee will offer a session on *Structural Materials in Cryogenic Design* on Wednesday. Also, the Refractory

Annual Meeting Abstracts

November 4th is the deadline for submission of abstracts to be considered for presentation in the IMD-sponsored sessions at the 1960 AIME Annual Meeting, which will be held from Feb. 19-23 in St. Louis.

In order to be considered for inclusion in the program, each submission must consist of:

1) A 200-300 word abstract, typed double-spaced on one side of the paper only. (No photographs, figures, tables, or diagrams, please.)

2) A separate information sheet containing the title of the paper and the name and address of *each* author.

3) Acknowledgements of research sponsorship, where *applicable*.

Abstracts and accompanying information should be submitted in 3 copies to:
The Metallurgical Society of AIME, IMD Program Committee, 29 W. 39th St., New York 18, N. Y.

Metals Committee has arranged a session on *Refractory Metal and Alloy Sheet Program.*

Thursday's *Refractory Metal Activities in Europe* has been arranged by the Refractory Metals Committee, and the Shaping and Forming Committee will offer a symposium on *Engineering Fundamentals of the Rolling Process.*

Breakfast meetings for authors and chairmen participating in each day's sessions are scheduled in the Constitution Room on Monday at 8:00 am and on Tuesday through Thursday at 7:30 a.m.

The Fall Meeting is being held in conjunction with the American Society for Metals. In addition to the technical sessions being held on Monday through Thursday, ASM is sponsoring a weekend seminar on Saturday and Sunday, Oct. 15 and 16, the subject of which is *Strengthening Mechanisms in Solids.* The seminar will be held at the Bellevue-Stratford Hotel.

Oxygen Steel Topic of Indian Meeting

Use of Oxygen in the Manufacture of Iron and Steel will be the subject of the 14th Annual Technical Meeting of The Indian Institute of Metals at Bhilai, Dec. 1-4. The Institute's Seventh Metallographic Contest and Exhibition also will be held during the Meeting. In addition, plant visits and social functions are included in the program.

Bhilai is the site of a Soviet-sponsored integrated steel plant mentioned in the November 1959 issue of JOURNAL OF METALS, p. 737.

For more information about the Meeting, contact: R. D. Lalkaka Honorary Secretary, The Indian Institute of Metals, 31 Chowringhee Road, Calcutta 16, India.

Commerce Dept. Offers Refractory Reports

A study on refractory metals and a report on 122 refractory materials have been released by the Office of Technical Services, Business and Defense Services Administration, US Dept. of Commerce.

The first report is *The Investigation of the Mechanism of Substructural Formation in Refractory Metals and the Relation to the Observed Mechanical Properties*—Part 1. A. Iannucci and others, Materials Research Corp., for Wright Air Development Center, USAF. Jan. 1960. 32 pages. (Order PB 161489 from OTS, US Dept. of Commerce, Washington 25, D. C., $1).

The other report is *Refractory Materials* (DMIC Memo 44). Defense Metals Information Center, Battelle Institute. Feb. 1960. 35 pages. (Order PB 161194 from OTS, US Dept. of Commerce, Washington 25, D. C. 50 cents).

AROUND THE SECTIONS

W. A. Wissler Gets Tone Medal Award

William A. Wissler has been named as the recipient of the Frank J. Tone Medal Award, which is awarded annually by the Niagara Frontier Section of AIME. The presentation will be made by Charles E. Emery, chairman of the Niagara Frontier Section at the Annual Award Meeting to be held on Nov. 16 in Buffalo.

The medal was established in 1956 by F. Jerome Tone and Franchot Tone to honor the memory of their father, Dr. Frank J. Tone, pioneer industrialist of the Niagara Frontier. It is awarded to a person who has made outstanding and important contributions to the science and practice of metallurgy. He must be a resident of the Niagara Frontier community.

Mr. Wissler retired in 1957 after 40 years of service with Haynes Stellite Co., and Union Carbide Corp. He attended Earlham College in Richmond, Ind., where he was born, and received his A.M. degree in 1914 from Indiana University. He instructed in chemistry at McGill University, Montreal, where under a fellowship he later worked on the smelting of lead, zinc, and gold ores.

Mr. Wissler joined Haynes Stellite Co. in 1917, and when this company was acquired by Union Carbide Corp. in 1921, he became an employee of that firm.

As a conusultant on casting operations to the Atomic Energy Commission, Mr. Wissler engineered and installed hot pressing facilities at the Haynes plant for fabricating shapes from tungsten and tungsten-carbide. This work was important to the production of Ordnance shell cores just subsequent to World War II and currently is finding applications for rocket nozzles for the Polaris and Minute Man missiles.

Mr. Wissler recently developed methods for manufacturing fine metal powders for powder metallurgy and powder spraying applications.

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Southern Ohio NOHC

The Southern Ohio Section of the National Open Hearth Committee will hold its annual Fall Meeting on Sept. 23-24, at the Deshler Hilton Hotel in Columbus, Ohio.

Welcoming remarks will be made by W. W. Bergmann, chairman of the Southern Ohio Section. This will be followed by a talk on *Safety With Oxygen* by George Dahm, Linde Co.

Technical papers presented during the Friday morning session will include *Production and Operating Problems with Roof Lance Furnaces, Bath Temperature Control on Oxygen Lance Furnaces, Method of Nozzle Setting,* and *Ladle Gunning.*

In the afternoon session *Quality of Aluminum Killed Steel Ingots* will be the title of one of the technical papers presented. Others will be *Pneumatic Injection of Graphite in Open Hearth Bath,* and *Removal of Slag and Refuse from Open Hearth Pits.* The session will close with *Progress Report on Basic Roofs.*

L. W. Moore, Wheeling Steel Corp., will be the toastmaster at the dinner which will be held at 7 p.m. The speaker will be Rev. Laurence Hall of St. Pauls Church, East Cleveland, Ohio.

An added attraction of the meeting will be the Ohio State—Southern Methodist football game which will be held in Columbus on Saturday, Sept. 24.

Conn. Section Meets Sept. 21

The Connecticut section will hold its fall meeting September 21, at the Chase Country Club in Waterbury. Dr. Charles H. Moore, newly appointed technical director of the Copper Product Development Association, will speak on *New Directions in the Copper Industry.*

Dr. Moore is the former director of corporate research and development for P. R. Mallory Co. He received degrees from the University of Virginia and Cornell, and was visiting professor at Rutgers for three years.

The dinner meeting is open to all interested people. Reservations should be made with Harold Cobb, Olin Mathieson Co., New Haven, Conn., or M. F. Burr, Chase Brass & Copper Co., Waterbury.

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Properties of Elemental and Compound Semiconductors

5 Sponsored by the Semiconductors Committee of the Institute of Metals Division, The Metallurgical Society, and the Boston Section of AIME. Boston, August 31-September 2, 1959

Edited by Harry C. Gatos

Contents

(Showing Part titles only: 21 papers were given, most followed by discussion.)
Metallurgical and Chemical Aspects
Solid State and Surface Reactions
Chemical and Lattice Defects
Physical Properties and Device Applications
The Role of Dislocations in Device Properties

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Proceedings of the 1960 Conference, Metallurgy of Elemental and Compound Semiconductors Boston, August 29-31, in preparation

6 **Flat Rolled Products II: Semi-Finished and Finished**
Proceedings of the Second Technical Conference Sponsored by the Mechanical Working Committee of the Iron and Steel Division, The Metallurgical Society, and the Chicago Section of AIME, Chicago, January 20, 1960

Edited by E. W. Earhart and R. D. Hindson

Contents

Hot Machine Scarfing of Semi-Finished Carbon Steels
Selection of Ingot and Slab Sizes for the Modern Strip Mill
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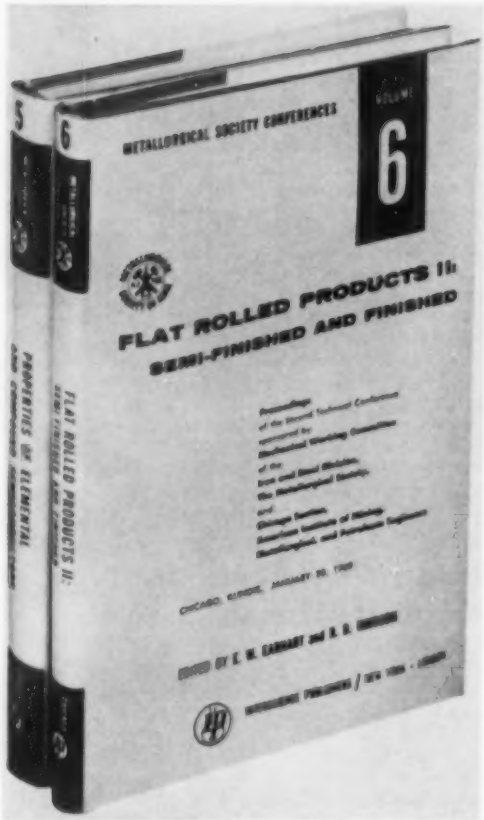

Previously published in the series

1 **Flat Rolled Products: Rolling and Treatment**—Chicago, January 21, 1959. Edited by T. E. Dancy and E. L. Robinson
148 pages $3.75

2 **Reactive Metals**—Buffalo, May 27-29, 1958. Edited by W. R. Clough
624 pages $15.00

3 **Quality Requirements of Super-Duty Steels**—Pittsburgh, May 5-6, 1958. Edited by R. W. Lindsay
320 pages $8.50

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408 pages $13.00

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Metallurgy of Columbium—Bolton Landing, New York, June 9-10, 1960. Edited by D. Douglass and F. W. Kunz
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Also to be published in cooperation with The Metallurgical Society of AIME

Extractive Metallurgy of Copper, Nickel, and Cobalt—New York, February 15-18, 1960. Edited by P. Queneau
Powder Metallurgy—New York, June 13-17, 1960. Edited by W. Leszynski
Agglomeration—Philadelphia, April 12-14, 1961. Edited by W. A. Knepper

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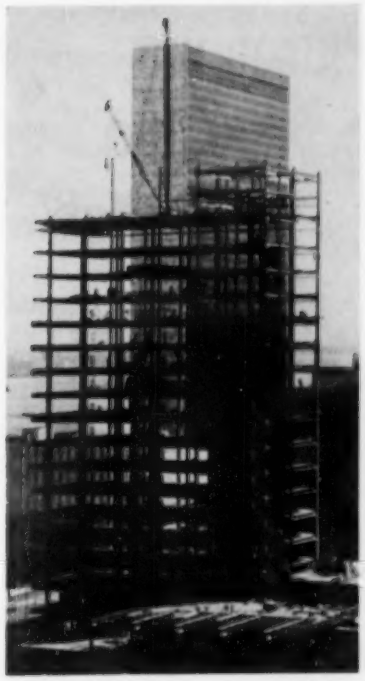
Engineering Center's Progress Marked by Topping Ceremony



With the last beam lowered into place on July 26th, the steel structure of the United Engineering Center was completed. Ceremonies were attended by representatives of the 19 societies which will occupy this building next July.

In keeping with tradition, a topping off ceremony took place. The American flag was raised along with the last and highest column, "in order to appease the wrath of the gods and to bring good luck to the new structure."

The new building will occupy an entire block on the west side of First Avenue from 47th to 48th Street. The 20-story steel framed all-welded structure is being built as headquarters for 18 leading American Engineering organizations.

There will be sixteen office floors in the building of which two floors will be unfinished for the present time, and the two top floors will have mechanical equipment. The Metallurgical Society will occupy the 13th and 14th floors.

The exterior of the new building is stainless steel and glass chosen by the architects for maximum beauty and strength. During the next few months curtain wall mullions and column covers of stainless steel will be installed. The basic curtain wall panel for the Center is 12½ ft high and 5 ft wide. Inside a rectangular framing of stainless steel is a sheet of window glass and a sheet of spandrel glass. These two glass sections are divided by a stainless steel bar.

In keeping with a completely modern outlook, the building will also be fully air-conditioned, contain high-speed elevators, modern lighting and the most up-to-date facilities, including a cafeteria.

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Officials of societies that will occupy space in the United Engineering Center gather for topping out ceremony. From left to right, O. B. Schier, II, Secretary, The American Society of Mechanical Engineers, E. L. Chandler, Treasurer, American Society of Civil Engineers, Beatrice Hicks, Society of Women Engineers, Alta Samuels, Society of Women Engineers, Andrew T. Boggs, Technical Secretary, American Society of Heating, Refrigerating, and Air-Conditioning Engineers, Inc., C. E. Davies, Executive Director, United Engineering Center, Andrew Fletcher, President, United Engineering Center, Ernest Kirkendall, Secretary, American Institute of Mining, Metallurgical and Petroleum Engineers, Inc., H. C. Cross, Executive Secretary, American Society of Heating, Refrigerating and Air-Conditioning Engineers and E. S. Hibabmen, Secretary, American Institute of Electrical Engineers.

Plutonium Volume Available

Extractive and Physical Metallurgy of Plutonium and Its Alloys is the title of the latest proceedings volume published by Interscience Publishers Inc., for The Metallurgical Society. The book is based on the Nuclear Metallurgy Committee symposium, held during the 1959 AIME Annual Meeting.

There is an introductory chapter written by W. D. Wilkinson entitled, aptly enough, Introduction to Plutonium Metallurgy. The Argonne National Laboratory engineer has also provided an invaluable 43-pp. annotated bibliography of publications on the subject.

The remainder of the contents is divided into extractive and physical metallurgy. Under extractive we find Alternative Routes for the Conversion of Plutonium Salts to Metals and Their Recovery Problems . . . Calcium Reduction of Plutonium Halides to Metal . . . Preparation of Metallic Plutonium . . . Distribution of Plutonium and Selected Impurity Elements Between Nitrate Solutions and Tri-n-butyl Phosphate . . . The Preparation of Plutonium Halides for Fused Salt Studies . . . and Removal of Fission Product Elements from Plutonium by Liquation.

Under physical metallurgy, the following papers are included: Some Principles of the Alloying Behavior of Plutonium . . . Plutonium-Cerium Phase Diagram . . . Plutonium-Zinc Phase Diagram . . . Metallography of Alpha Plutonium . . . The Etching of Plutonium and Its Alloys by Cathodic Bombardment . . . A Study of the Transformation Kinetics of Alpha, Beta, and Gamma Plutonium . . . Some Experiments in Zone Refining Plutonium . . . and Behavior of Some Delta-Stabilized Plutonium Alloys at High Pressures.

Discussions follow many of the papers in this 314-pp. book.

Price of the volume is $10.50; however, AIME members may have it for the 20 pct discount price of $8.40. Nonmembers should order directly from Interscience Publishers Inc., 250 Fifth Ave., New York 1, N. Y. Members should order through The Secretary, The Metallurgical Society of AIME, 29 West 39th St., New York 18, N. Y.

NEW BULLETIN

A new bi-weekly bulletin of "Engineers Available" is now being published by the Engineering Societies Personnel Service, Inc. Distributed free of charge, the bulletin contains synopses of the experience of engineers who have registered with the Service and are seeking a new position.

Any employer interested in receiving the bulletin should so advise E.S.P.S. at 8 W. 40th St., New York 18, N.Y., and your name will be placed on the mailing list. Any engineer who is registered with the Service or wishes to register is entitled to a 35-word notice in the bulletin. Write to the New York office for forms. Your qualifications will be brought to the attention of employers on our mailing list without revealing your identity.

It is planned to extend the publication to the midwestern and western offices in the near future.

Change of Address

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Total AIME members on June 30, 1960, was 33,844; in addition 2,514 Student Members were enrolled.

ADMISSIONS COMMITTEE

W. L. Brytczuk, Chairman; S. C. Carapella, Jr., J. W. Hanley; T. D. Jones; H. C. Larson; Harold Margolin; Shadburn Marshall.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

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Muska, George J., Pittsburgh, Pa.
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Kaufman, Sidney F., Pittsburgh, Pa.

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Imperial College, London
Howard, Richard A.
Purdue University
Darda, Gerald E.
Rice University
Hartman, David E.

HOWARD G. SATTERTHWAITE

An Appreciation by
James F. Smith
George T. Hanson

Howard G. Satterthwaite died May 11, 1960. The word of his sudden death at his home in Escondido, Calif., brought expressions of deep sympathy and regret from his many friends and business associates, who affectionately knew him as *Satt*.

Satt's Anaconda career was marked by many outstanding achievements in metallurgical operations and in the administration field. He played an important part in the Company's venture in the production of magnesium during World War II, and later in Anaconda's entry into the aluminum industry. As manager of the aluminum plant in Columbia Falls, Mont., he endeared himself to all his co-workers with his fair-mindedness, boundless enthusiasm, and capable leadership.

Born in Girard, Kans., April 18, 1891, Satt graduated from Washington State College with a B.S. in 1915, and a year later began his career with Anaconda as a laborer in the Company's experimental zinc plant in Anaconda, Mont.

In December 1916, he was transferred to the newly-constructed electrolytic zinc plant in Great Falls, Mont. Here he worked his way from a laborer to shift foreman, general foreman, assistant superintendent, and superintendent. He returned to the city of Anaconda in 1927 as superintendent of the zinc plant there, and was transferred back to Great Falls 10 years later to become assistant general superintendent of the entire reduction plant.

During World War II, Satt was selected to lead one of Anaconda's contributions to the war effort as manager of Basic Magnesium Inc., a magnesium reduction plant in Henderson, Nev. After the war and completion of the Henderson job, Satt returned to Great Falls.

Shortly thereafter, Anaconda called upon Satt to do some more pioneering, and in November 1952 he undertook the job of selecting an aluminum reduction process for the new Columbia Falls location. He was named manager of the aluminum plant during the construction period, and guided the plant's first three years of operation before his retirement on July 1, 1958.

His association with AIME dated from 1942, and he took an active part in the Montana section meeting schedule.

EUGENE GIFFORD GRACE

An Appreciation

Eugene Gifford Grace, member of the American Institute of Mining, Metallurgical and Petroleum Engineers since 1912, left at his death a rich legacy—the example of a life lived in successful achievement of great purposes.

Endowed with rare qualities of intellect and a capacity for inspiring leadership, he devoted his long career to developing Bethlehem Steel into the world's second largest steel producer and largest shipbuilder.

He was a supporter of research that led to steadily improving methods and products. And he was a leader in bringing about harmonious employer-employee relations in his own company and in the steel industry.

Mr. Grace was graduated in 1899 from Lehigh University as an electrical engineer, going immediately to work for Bethlehem. From his first job as a crane operator, he rose to general manager in 1908 and president in 1916. After long serving as Bethlehem's chief executive officer, he continued in that capacity following his election as chairman in 1945. He became honorary chairman in 1957. His death occurred July 25, 1960, in his 84th year.

He was accorded numerous honors during his lifetime, including the AIME Rand Medal, as well as the Gary Medal and the Bessemer Medal.

The JOURNAL OF METALS is proud to record here its appreciation of Mr. Grace and of his many contributions to the steel industry.

OBITUARIES

William J. Reilly (member, 1955) died on July 9th. At his death, Mr. Reilly was general manager of the Steel div., Ford Motor Co. He started with Ford as asst. superintendent of the steel division in 1946. Prior to this he was with the Youngstown Sheet & Tube Co.

NECROLOGY

Date Elected	Name	Date of Death
1927	Bateman, Glen L.	Jan. 24, 1960
1910	Crease, H. H.	Unknown
1914	Dickenson, E. H.	June 8, 1960
1948	Duffy, Edward J.	Nov. 15, 1958
1910	Fujita, Heitaro	Unknown
1917	Gilman, G. H.	June 28, 1960
1939	Johnson, F. E.	March 15, 1960
1956	McCulloch, R. A.	March 8, 1959
1901	Moses, Horace (Legion of Honor)	June 20, 1960
1955	Reilly, W. J.	July 9, 1960
1914	Wright, L. Ira	June 16, 1960
1940	Yanez, Jose R.	Unknown

Coming Events

Sept. 15-16, Eighth Annual Engineering Management Conference, Morrison Hotel, Chicago.

Sept. 19-20, 58th Fall Meeting, Steel Founders' Society of America, The Homestead, Hot Springs, Va.

Sept. 23-24, Southern Ohio Section, NOHC, 24th Annual Technical Conference, Deshler, Hilton Hotel, Columbus.

Oct. 3, Chicago Section, NOHC, Fall Dinner Meeting, Phil Smidt's Restaurant, Hammond, Ind.

Oct. 5-7, Rocky Mt. Minerals Conference, Newhouse Hotel, Salt Lake City.

Oct. 12, Annual Meeting of the Cast Bronze Bearing Institute, Grove Park Inn, Asheville, N. C.

Oct. 12-13, Seventh National Symposium, American Vacuum Society, Cleveland-Sheraton Hotel, Cleveland.

Oct. 12-14, Fourth Conference on Analytical Chemistry in Nuclear Reactor Technology—First Conference on Chemistry in Nuclear Reactor Technology, Gatlinburg, Tenn.

Oct. 13-14, Southwestern Section, NOHC, Fall Meeting, Statler-Hilton Hotel, Dallas.

Oct. 13-15, 1960 Annual Meeting of the Non-Ferrous Founders' Society, Grove Park Inn, Asheville, N. C.

Oct. 17-18, Annual Convention, The Magnesium Association, Pick Carter Hotel, Cleveland.

Oct. 17-20, Fall Meeting of The Metallurgical Society of AIME, Sheraton Hotel, Philadelphia.

Oct. 17-21, Fall Meeting of the Societe Francaise de Metallurgie, Maison de la Chimie, Paris.

Oct. 23-26, Seminar on Rare Earth Research, Arrowhead Center, University of California, Los Angeles.

Oct. 28, Eastern Section, NOHC, 14th Annual Conference, Warwick Hotel, Philadelphia.

Nov. 4, Pittsburgh Section, AIME, 15th Annual Off-the-Record Meeting, Penn-Sheraton Hotel, Pittsburgh.

Nov. 8-11, First National Die Casting Exposition and Congress, Detroit Artillery Armory, Detroit.

Nov. 14-16, Fifteenth Technical and Operating Conference, Steel Founders' Society of America, Hotel Carter, Cleveland.

Nov. 30-Dec. 2, AIME 18th Electric Furnace Conference, Morrison Hotel, Chicago.

Jan. 18, 1961, Conference on Bar and Allied Products, sponsored by Mechanical Working Committee of The Metallurgical Society, Penn-Sheraton Hotel, Pittsburgh.

Feb. 26-March 2, 1961, AIME Annual Meeting, Chase & Park Plaza Hotels, St. Louis, Mo.

Mar. 13-14, 1961, 59th Annual Meeting, Steel Founders' Society of America, Drake Hotel, Chicago.

Apr. 10-12, 1961, 44th National Open Hearth Steel Conference and Blast Furnace, Coke Oven and Raw Materials Conference, Sheraton Hotel, Philadelphia.

Apr. 26-27, 1961, Cleveland Section, The Metallurgical Society, Technical Conference on High Temperature Materials, Hotel Carter, Cleveland.

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THE FALL MEETING OF THE METALLURGICAL SOCIETY

Program and Abstracts

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and
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General Information

Admission to Sessions: Only those persons who have registered for the Fall Meeting of The Metallurgical Society will be admitted to technical sessions sponsored by the Society and held at the Sheraton Hotel.

Registration: *Avoid the rush* if you arrive for the meeting before Monday by registering on Sunday evening. The registration desk will be located in Parlors A and B in the Sheraton Hotel. Hours as follows: Sunday, 3:00 to 9:00 pm; Monday through Wednesday, 8:00 am to 4:00 pm; Thursday, 8:00 am to 3:00 pm.

Registration fees:

AIME Member	$5.00
AIME Student Member	No fee
Nonmember	$10.00
Nonmember Student	$1.00

Authors' and Chairmen's Breakfasts: Breakfast meetings for authors and chairmen participating in each day's sessions are scheduled as follows: Monday at 8:00 am, Tuesday through Thursday at 7:30 am, all in the Constitution Room.

Copies of papers: In general, copies of individual papers presented at the Fall Meeting are not available from The Metallurgical Society. Exceptions exist in the cases where symposiums are published in a unit, such as the Nuclear Metallurgy series. Notice of the availability of such publications ordinarily appears in meeting programs and the Journal of Metals as well as other periodicals in related fields. Authors are at liberty, of course, to distribute copies of their own papers; therefore, it is suggested that persons wishing to obtain such copies contact authors directly.

Summary of ASM Technical Sessions

Monday, Oct. 17

All day — ASM - AEC Conference—Non - oxide Compounds for Nuclear Fuels

9:00 am — Materials for the Aerospace Age
Physical Metallurgy I

2:00 pm — Metal Joining
Physical Metallurgy II

(Continued on next page)

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(Summary of ASM Sessions, Continued)

Tuesday, Oct. 18

9:00 am — Failure Analysis I
Phase Diagrams
Steel I

2:00 pm — Failure Analysis II
Stainless Steels
Steel II
Plastic Deformation

Wednesday, Oct. 19

2:00 pm — Recent Advances in Metallurgical Microscopy
Steel III
High Temperature Materials

Thursday, Oct. 20

9:00 am — Effects of Structure on Properties I
Nuclear Materials

2:00 pm — Effects of Structure on Properties II
Embrittlement

Condensed Program - Aime Technical Sessions, Sheraton Hotel, Philadelphia

	West Ballroom	East Ballroom	Pa. Room—East	Pa. Room—West	Hall of Flags
Monday, Oct. 17, 9:00 am	Metallurgy in the Army	Thermodynamics, Phase Equilibria, and Structure	Recent Advances in the Metallurgy of Stainless Steel (Symposium)	Ingot Structure Control I (Symposium)	Diffusion (Symposium)
Monday, Oct. 17, 2:00 pm	Metallurgy in the Army	Observation of Imperfections, Grains, and Surfaces	Recent Advances in the Metallurgy of Stainless Steel (Symposium)	Ingot Structure Control II (Symposium)	Diffusion
Tuesday, Oct. 18 9:00 am	Titanium (to 10:30 am)	Phase Transformations (to 10:30 am)		Process Simulation (Symposium)	Effects of Extremely High Pressures (Symposium)
Tuesday, Oct. 18, 11:00 am	Institute of Metals Lecture				
Tuesday, Oct. 18, 2:00 pm	Titanium Versus Steel and Aluminum in Pressure Vessels for Hydrospace, Aerospace, and Space (Symposium)	Mechanical and Metallurgical Principles Affecting Hot Extrusion (Panel Discussion)		Theory and Application of Physical Chemistry (Symposium)	Effects of Extremely High Pressures
Wednesday, Oct. 19, 9:00 am	Diffusion Controlled Processes (to 10:30 am)	Symposium on Metallic Moderators and Cladding Materials	Effects of Gases in Metals (to 10:30 am)	The Decomposition of Austenite by Diffusional Processes (Symposium)	Physical, Electrical, and Magnetic Properties of Metals and Alloys (to 10:30 am)
Wednesday, Oct. 19, 2:00 pm	Refractory Metal and Alloy Sheet Program (Symposium)	Symposium on Metallic Moderators and Cladding Materials	Structural Materials in Cryogenic Design (Symposium)	The Decomposition of Austenite by Diffusional Processes (Symposium)	Plastic Deformation I
Thursday, Oct. 20 9:00 am	Refractory Metal Activities in Europe (Symposium)	Plastic Deformation II	Powder Metallurgy I	Engineering Fundamentals of the Rolling Process (Symposium)	Precipitation and Dispersed Phases
Thursday, Oct. 20, 2:00 pm	Refractory Metals	Creep, Fatigue, and Fracture	Powder Metallurgy II	Textures, Recrystallization, and Recovery	

Special Events

Army Day Luncheon: On Monday, October 17, the opening day of the Fall Meeting, the Institute of Metals Division will sponsor a symposium on Metallurgy in the Army—Requirements, Applications, and Research. This is the second in a series of armed services presentations intended to acquaint those engaged in nonmilitary research and development activities with the scope and extent of corresponding military activities.

In conjunction with the symposium, there will be an open luncheon featuring a talk by John R. Townsend of the Office of the Director of Defense Research and Engineering entitled "The Role of Metals in National Defense." Luncheon tickets, priced at $3.75, **must be purchased before 9:30 a.m. Monday** at the Registration Desk.

Institute of Metals Lecture: Formerly known as the IMD Annual Lecture and customarily presented at the Annual Meeting, the Institute of Metals Lecture will be given at the Fall Meeting this year. Entitled *The Work Hardening of Metal Crystals*, the lecture will be given by Professor N. F. Mott, F.R.S., of Cavendish Laboratory in the Department of Physics, Cambridge University, Cambridge, England, beginning at 11:00 a.m. on Tuesday, October 18, in the West Ballroom of the Sheraton Hotel.

Annual Fall Dinner: The annual Fall Dinner of the Society will be held in the Pennsylvania Room of the Sheraton Hotel on Tuesday, October 18, at 7:30 p.m. Preceding it at 7:00 p.m. will be an open cocktail party in the Constitution Room. Ladies are welcome at both events, of course. Dress is informal (business suits).

Dinner tickets are priced at $6.50 and will be on sale at the Registration Desk. In order that adequate facilities may be provided, registrants are urged to buy their dinner tickets as early as possible.

Powder Metallurgy Luncheon: This luncheon, which is open to all who wish to attend, is a traditional activity for powder metallurgists attending the Fall Meeting. Noted for its informality, the luncheon is conveniently scheduled between morning and afternoon technical sessions on Thursday, affording an excellent opportunity to renew old friendships and make new ones in a most pleasant atmosphere. Tickets priced at $4.00 will be on sale at the Registration Desk throughout the meeting. Because of the popularity of this event, it is advisable that you purchase tickets early.

National Metal Congress: The Fall Meeting is being held in conjunction with the National Metal Congress sponsored by the American Society for Metals. In addition to the technical sessions being held on Monday through Thursday (see summary, pages 2 and 3), ASM is sponsoring a weekend seminar on Saturday and Sunday, October 15 and 16, the subject of which is *Strengthening Mechanisms in Solids*. Both the seminar and the technical sessions will take place at the Bellevue-Stratford Hotel.

CALENDAR OF EVENTS

Sunday, October 16

3:00 to 9:00 pm	Registration (Parlors A and B)
4:30 pm	IMD Program Committee Meeting (Connie Mack Room)
5:30 pm	IMD Scope Committee Meeting (Connie Mack Room)
7:00 pm	Met. Soc. Committee on Metallurgical Profession Meeting (Room 556)

Monday, October 17

8:00 am	Breakfast for Authors and Session Chairmen (Constitution Room)
8:00 am to 4:00 pm	Registration (Parlors A and B)
12:00 noon	IMD Chemistry and Physics of Metals Committee Meeting (Place to be announced)
12:15 pm	Army Day Luncheon (East Ballroom)
3:30 pm	IMD High-Temperature Alloys Committee Meeting (Independence Room)
4:00 pm	IMD Publications Committee Meeting (Connie Mack Room)
5:00 pm	IMD Ferrous Metallurgy Committee Meeting (Parlors C and D)

Tuesday, October 18

7:30 am	Breakfast for Authors and Session Chairmen (Constitution Room)
8:00 am to 4:00 pm	Registration (Parlors A and B)
8:30 am	IMD Structural Materials Committee Meeting (Independence Room)
9:00 am	Met. Soc. Publications Committee Meeting (Room 547)
9:00 am	IMD Non-Ferrous Metallurgy Committee Meeting (Parlors C and D)
9:00 am	IMD Corrosion Resistant Metals Committee Meeting (Connie Mack Room)
11:00 am	Institute of Metals Lecture (West Ballroom)
12:00 noon	Yale Metallurgical Alumni Luncheon (Room 548)
12:00 noon	IMD Executive Committee Luncheon Meeting (Constitution Room)
4:00 pm	IMD Membership Committee Meeting (Independence Room)
4:00 pm	IMD Melting and Casting Committee Meeting (Connie Mack Room)
4:30 pm	IMD Physical Metallurgy Committee Meeting (Parlors C and D)
5:00 pm	IMD Refractory Metals Committee Meeting (Room 527)
7:00 pm	Cocktail Party for Dinner Guests (Constitution Room)
7:30 pm	Annual Fall Dinner (Pennsylvania Room)

Wednesday, October 19

7:30 am	Breakfast for Authors and Session Chairmen (Constitution Room)
8:00 am to 4:00 pm	Registration (Parlors A and B)
12:00 noon	IMD Titanium Committee Luncheon Meeting (Room 548)
12:00 noon	IMD Powder Metallurgy Committee Executive Group Meeting (Place to be announced)
12:30 pm	Metallurgical Society Board of Directors Luncheon Meeting (Constitution Room)
4:00 pm	IMD Nuclear Metallurgy Committee Meeting (Connie Mack Room)

Thursday, October 20

7:30 am	Breakfast for Authors and Session Chairmen (Constitution Room)
8:00 am to 3:00 pm	Registration (Parlors A and B)
12:00 noon	IMD Shaping and Forming Committee Luncheon Meeting (Room 548)
12:15 pm	Open Luncheon Sponsored by IMD Powder Metallurgy Committee (Tiered Balcony)

Monday, October 17

Metallurgy in the Army
Requirements, Applications, and Research

9:00 am to 12:00 m, West Ballroom

(Sponsored by Institute of Metals Division)

Chairmen: J. L. Martin, Ordnance Materials Research Office, and David Swan, Union Carbide Metals Co.

R & D: Shield for the Future: Lt. General A. G. Trudeau, Chief of Research and Development, Department of the Army.

Technical Session: Metallurgical and Related Requirements and Applications.

Combat and Tactical Vehicles: C. B. Salter, U. S. Army Ordnance Tank-Automotive Command.

Communications: S. B. Levin, Deputy Director, Institute for Exploratory Research, U. S. Army Signal Corps.

Solid Propellant Rocket Motors: J. L. McDaniel, Technical Director, Research and Development Operations, U. S. Army Ballistic Missile Agency.

Thermodynamics, Phase Equilibria, and Structure

9:00 am to 12:00 m, East Ballroom

(Based on Submission of Research Abstracts)

Chairmen: J. B. Newkirk, General Electric Co. and S. K. Nash, Frankford Arsenal

Thermodynamics of Solid Iron: P. D. Anderson and Ralph Hultgren, University of California

Heat contents of extremely pure iron have been measured from room temperature to 1160°C in a diphenyl ether calorimeter. Three samples were used, from widely different sources and with different trace impurities. Results agreed with one another within experimental precision and with higher temperature heat contents recently published by Olette and Ferrier. An enthalpy table room temperature to the melting point of iron has been prepared, the first such table of high precision based entirely on experimental data. Heat contents obtained by integrating true C_p measurements found in the literature agree well at lower temperatures but diverge widely from one another and from the table at high temperatures. The table differs significantly from quantities derived by Darken and Smith and others from calculation on phase equilibria. It was shown that reasonable extrapolations of the enthalpy of γ-Fe to room temperature may differ by as much as 700 cal per g-atom; such extrapolations should be of limited value in a themodynamic analysis of the formation of martensite. The properties of a hypothetical non-magnetic α iron are also considered.

This work was made possible by financial support from the American Iron and Steel Institute.

Statistical Description of Distribution of Phases: D. E. Gücer, Brown University.

An attempt has been made to apply the theory of runs of mathematical statistics together with the point count method to the quantitative description of the state of distribution of phases in a multi-phase alloy.

It has previously been shown that the point count method adequately gives the volume, area, and line fractions of individual phases. However, if the order in which the points fall into the phases is recorded, additional information regarding the distribution of phases can be derived by the use of the theory of runs.

If all possible distributions of points are given equal weight, the distribution with the highest probability would be expected to occur most frequently. If any distribution other than this occurs, this is evidence of the presence of assignable causes which may be physical, chemical, or mechanical in nature. Such causes change the distribution in the direction of dispersion or segregation. Consequently, three states of distribution are distinguished on the basis of either the number of runs or the length of the runs: segregated, random (most probable) and dispersed. The theory can be applied to the individual phases as well as to the system as a whole.

Statistical significance tests have been proposed for the statements on distribution. A statistical distribution index has been devised to determine the degree of segregation or dispersion.

This work has been supported by the Metallurgy and Materials Branch, Division of Research, U.S. Atomic Energy Commission.

Phase Diagram of the Pseudo-binary System Bi_2Te_3-Sb_2Te_3: J. R. Wiese and Richard Pyle, Franklin Institute Laboratories.

The semiconductor compounds Bi_2Te_3 and Sb_2Te_3 are isomorphous, with a rhombohedral (C33) structure. X-ray analysis, using powder techniques, indicates these compounds are soluble in each other in all proportions. Cooling curves were obtained at 10 mole pct composition intervals by conventional thermal analysis techniques and indicate that the liquidus and solidus of the system are very nearly congruent. The solidus compositions were confirmed by polarographic analysis. Thermal expansion data is also presented.

This work has been sponsored by The Thermoelectric Effects Research Cooperative Program.

Structural Study of the Compound $AgSbTe_2$: R. W. Armstrong, J. W. Faust, Jr., and W. A. Tiller, Westinghouse Electric Corp.

X-ray, optical and electron microscope, and chemical etching techniques have been used to investigate a striated microstructure observed in zone refined single crystals of the $AgSbTe_2$ structure. This microstructure, originally thought to be fine twin lamellae, has been identified as a Widmanstatten precipitate of Sb_2Te_3 upon the {111} planes of the matrix fcc material. In addition, the evidence obtained from all the measurements made on this material indicates that the ternary compound of the $AgSbTe_2$ type does not melt congruently.

Miscible Laves-type Compounds in Ternary Systems. J. B. Darby, Jr., Argonne National Laboratory.

The pseudobinary sections between known AB_2 phases composed of transition elements and having a Laves-type structure have been investigated at 900°C in 20 ternary systems using optical and X-ray metallography. The selected systems conform to the following restrictions:

1. The A-elements are from Groups IVB and VB.
2. The B-element is common to both component compounds and is a transition element of the first or third long period and from Group V through Group VIII.
3. Both component compounds have the same structure type, either C14 or C15.

A complete range of miscibility between the binary Laves-type compounds was found in the majority of the systems investigated. The stability of C14 and C15 compounds will be discussed in terms of size and electronic considerations.

This abstract is based on work performed at the Argonne National Laboratory under the auspices of the U.S. Atomic Energy Commission.

Isothermal Section of the Cr-Zr-O System at 1200°C. Chung-kong Hwu and Michael Hoch, University of Cincinnati.

The isothermal section of the Cr-Zr-O system at 1200°C was investigated using high temperature and room temperature powder X-ray diffraction and micrometallographic methods. The samples were prepared by mixing the metal powders and their oxides and heating to 1200°C under high vacuum until equilibrium was obtained. The system showed one ternary compound, Cr_2Zr_2O with the ϵ type structure and lattice parameter of $a = 11.957 \pm 002$ Å.

The homogenity range of the single phase regions in the ternary system is very narrow, the β Zr (bcc) being the largest, extending from 3 atomic pct O in the Zr-O binary to 6 atomic pct Cr in the Zr-Cr binary. Therefore, the diagram consists of large three-phase regions, which are as follows:

(β = cubic, α = hexagonal, t = tetragonal)

$Cr + Cr_2O_3 + t\text{-}ZrO_2$
$Cr + ZrCr_2 + t\text{-}ZrO_2$
$ZrCr_2 + Zr_3Cr_2O + t\text{-}ZrO_2$
$ZrCr_2 + \alpha\text{-}Zr + \beta\text{-}Zr$
$ZrCr_2 + Zr_3Cr_2O + \alpha\text{-}Zr$
$\alpha\text{-}Zr + Zr_3Cr_2O + t\text{-}ZrO_2$

This research was supported in part by the Aeronautical Research Laboratory WADD, Air Research & Development Command, U.S.A.F.

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Zirconium-rich Corners of the Ternary Systems Zr-Co-O and Zr-Ni-O: M. V. Nevitt and J. W. Downey, Argonne National Laboratory.

The approximate phase boundaries for the 950°C isothermal sections in the ternary systems Zr-Co-O and Zr-Ni-O have been determined for the composition range from 50 to 100 atomic pct Zr. The two systems exhibit phase relations which are similar in the following respects:

1. There are no extensive solid solution phase fields.
2. Ternary phases based on Zr_3B stoichiometry for the metal components occur in both systems with variable O content from about 5 to 20 atomic pct.

The two ternary phases give essentially identical Debye-Scherrer patterns but their structure has not been identified.

The binary phase Zr_2Co, whose existence has been previously reported, can be indexed from powder patterns as a tetragonal structure with the following lattice parameters: $a_o = 6.367 \pm 0.002$A, $c_o = 5.513 \pm 0.002$A. It is apparently a $CuAl_2$-type (C16) structure, isomorphous with Zr_2Ni and Hf_2Ni. The frequency of occurrence of the $CuAl_2$-type structure and another large family of A_2B phases, the Ti_2Ni-type, is discussed in terms of an atomic size effect. Values of the ratio R_A/R_B, where R_A and R_B are the C. N. 12 atomic radii, fall between 1.09 and 1.54 for $CuAl_2$-type phases, but are restricted to the narrower range 1.05 to 1.27 for Ti_2Ni-type phases.

W. L. Larsen, D. E. Williams and W. H. Pechin: Crystallographic and Phase Relationships of the Zirconium-Cobalt System, Ames Laboratory Report No. IS-17, Iowa State University of Science and Technology, November 1959, p. 92.

J. F. Smith, M. E. Kirkpatrick, D. M. Bailey and D. E. Williams: Crystallographic and Phase Relationships of the Nickel-Zirconium and Nickel-Hafnium Systems, Ames Laboratory Report No. ISC-1050, Iowa State University of Science and Technology, March 1959, p. 55.

D. K. Deardorf: Hafnium Phase Diagrams, U.S. Bureau of Mines Report No. USBM-U-623, July 1959, p. 29.

D. K. Deardorf: Private Communication.

This abstract is based on work performed at the Argonne National Laboratory under the auspices of the U.S. Atomic Energy Commission.

Occurrence of AB_5 Compounds in Binary and Ternary Transition Element Systems: A. E. Dwight, Argonne National Laboratory.

A study was made of the occurrence of AB_5 compounds in which A and B are transition elements lying to the left and right, respectively, of the Mn group. Compounds were investigated which have the following prototypes: $TiNi_5$, $MgCd_5$, $TiCu_5$, Cu_5Au, $TiAl_5$, $ZrAl_5$ and $AlFe_5$. For the first three, which possess variants of the cph structure, relations between the c/a ratio and atomic radii are discussed. The latter four exhibit variants of a cubic structure. The influence of the radius ratio was studied for the seven crystal structure types. The occurrence of the various crystal structure types is related to the position in the periodic table of the A and B elements. The alloying behavior of the A elements Th, and U, compared with members of the Sc and Ti groups, indicates that Th and U more closely resemble the Ti group elements than the Sc group elements. Pseudo-binary sections were constructed between a number of AB_5 compounds, in particular between various ANi_5, APd_5 and APt_5 compounds.

This work was performed under the auspices of the U.S. Atomic Energy Commission.

Effects of Annealing on the Structure of Pyrolytic Graphite: E. R. Stover, General Electric Co.

Pyrolytic graphite deposited from methane between 1700° and 2500°C contains sheets of carbon atoms arranged as in the basal planes of graphite. X-ray analysis reveals little initial crystalline order between layers, and average layer separation is about 2 pct greater than in graphite. Electron microscope observations have shown that the sheets consist of small crystals separated by tilt boundaries; these are arranged in spherical zones which also intersect in tilt boundaries. The maximum size and radii of curvature of the zones, and the angles of tilt at the boundaries are important features of the microstructure.

Upon heating in the range 2500° to 3000°C, the tilt boundaries tend to straighten. This process has been studied in a variety of pyrolytic graphite deposits heated to successively higher temperatures. Changes in dimensions, physical properties, and microhardness have been correlated with changes in structure as revealed by X-ray diffraction and both optical and electron metallography. There is a net expansion parallel to the surface of deposition and a larger contraction perpendicular to this plane. There is also a reduction in hardness as a crystal structure characteristic of perfect graphite is approached.

Straightening of the layers is restrained by certain features of the microstructure. Pyrolytic deposits which contain a greater number of regions having high misorientation between crystals undergo dimensional change and softening at higher temperatures of annealing. It is suggested that a contribution to the driving force for annealing results from the compressive stress developed perpendicular to the layers at dihedral angles upon heating, due to the anisotropy of thermal expansion in graphite.

This work was sponsored by the Hanford Atomic Products Operation of the General Electric Company, and supported by the U.S. Atomic Energy Commission.

Recent Advances in the Metallurgy of Stainless Steel

9:00 am to 12:00 m, Pennsylvania Room-East

(Sponsored by IMD Corrosion Resistant Metals Committee)

Chairmen: S. A. Rosenberg, National Bureau of Standards, and L. R. Scharfstein, The Carpenter Steel Co.

Mechanism of Hardening in 17-7 PH Stainless Steel: E. E. Underwood, A. E. Austin, and G. K. Manning, Battelle Memorial Institute

Influence of Carbides on the Transformation Behavior of Semi-austenitic Stainless Steels: G. N. Aggen and R. A. Lula, Allegheny Ludlum Steel Corp.

Quantity and Form of Carbides in Austenitic and Precipitation Hardening Stainless Steels: R. J. Bendure, L. C. Ikenberry, and J. H. Waxweiler, Armco Steel Corp.

Submicroscopic Structures in Tempering of 410 Stainless Steels: J. J. Hauser, J. M. Capenos, and B. R. Banerjee, Crucible Steel Corp. of America

Dependence of Creep Rupture on Creep Behavior: F. Garofalo, R. W. Whitmore, W. F. Domis, and F. von Gemmingen, U. S. Steel Corp.

Ingot Structure Control I

9:00 am to 12:00 m, Pennsylvania Room-West

(Sponsored by IMD Melting and Casting Committee)

Chairmen: M. C. Flemings, Massachusetts Institute of Technology, and T. E. Leontis, Dow Metal Products Co.

Quantitative Treatment of Ingot Structure Control: W. A. Tiller, Westinghouse Electric Corp.

The unidirectional freezing of a semi-infinite liquid from one end has been treated by calculating the solute and temperature distributions with position and time ahead of the solid-liquid interface which advances at a rate proportional to $(\text{time})^{-1/2}$. The nucleation probability for new grain formation is calculated as a function of the constitutional and thermal properties of the alloy, the pouring conditions and the catalytic efficiency of the nucleating agents. A transition from columnar to equiaxed crystal formation can be determined. The grain sizes in these two zones of the ingot has also been calculated. The effect of a finite ingot has also been considered and in such an ingot the end-effects become very important. The theoretical predictions are in good agreement with experiment.

Solidification in Undercooled Metals: J. L. Walker, General Electric Co.

The specific problem in this presentation is the determination of the dependence of the grain size of nickel on the undercooling at which crystallization is initiated. This dependence has been the subject of considerable speculation among metallurgists. Because of the difficulty encountered when an attempt is made to undercool large samples of metal, very little information has been

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available pertaining to grain size in undercooled metals.

A technique for undercooling large samples of nickel has been developed. This technique was used to prepare the specimens selected for study in this investigation.

The grain size is reported and explained where possible in terms of existing theory of nucleation and growth of crystals in a melt. The results show a marked decrease in grain size at a critical undercooling. This behavior suggests a marked increase in the ratio of the effective rate of nucleation to that of growth. Preliminary measurements of the freezing velocity at large undercoolings are described and, where possible, related to the grain size observed. An hypothesis is presented suggesting a relationship between the number of grains that nucleate and a mechanical disturbance observed to occur when nickel crystallizes at large undercoolings. The results indicate that pressure fluctuations in an undercooled melt can produce significant changes in grain size.

Segregation in Alloy Ingots: J. W. Holladay, Battelle Memorial Institute

The theory of freezing segregation of alloys is reviewed. The factors affecting segregation during normal freezing are discussed for a simplified, idealized case of a progressively freezing bar of metal. The theory is then applied to the various stages of freezing of an alloy ingot cast in a mold. Segregation in the chilled layer, the columnar zone and equiaxed zone is considered. Factors affecting segregation include (1) the type of alloying addition or impurity, (2) mode of crystallization, (3) temperature gradient, (4) mixing in the liquid metal, and (5) rate of solidification. Segregation is related to specific alloys by the distribution coefficient, i.e., ratio of the concentration of alloying element in the solid to its concentration in the liquid, and to the slope of the liquidus curve of the binary phase diagram. The tendency for segregation to occur in iron, titanium, and other alloy ingots are discussed. The formation of oxide and sulfide films and non-equilibrium precipitates are explained by segregation theory.

Diffusion

9:00 am to 12:00 m, Hall of Flags

(Sponsored by IMD Chemistry and Physics of Metals Committee)

Chairmen: C. A. Wert, University of Illinois, and *R. A. Swalin, University of Minnesota.*

Diffusion in Magnetic Materials: C. E. Birchenall, University of Delaware.

Self-diffusion in Silver-Gold Alloys: L. Slifkin, University of North Carolina, and W. Mallard, Emory University.

Effect of Simultaneous Plastic Strain on Diffusion in Metals: R. Balluffi, University of Illinois, J. Darby, Argonne National Laboratory, and C. Tomizuka, University of Arizona.

Army Day Luncheon

Monday, October 17

12:15 pm — East Ballroom

Speaker: John R. Townsend, Office of the Director of Defense Research and Engineering

Topic: The Role of Metals in National Defense

Luncheon tickets, priced at $3.75, **must be purchased before 9:30 am** at the Registration Desk.

Metallurgy in the Army
Requirements, Applications, and Research

2:00 to 5:00 pm, West Ballroom

Chairmen: J. L. Martin, Ordnance Materials Research Office, and *David Swan, Union Carbide Metals Co.*

Introductory Remarks: Brig. Gen. W. J. Ely, Director, Army Research Office.

Metallurgical Research in the Army: Lt. Col. L. G. Klinker, Chemical and Materials Branch, Physical Sciences Division, Army Research Office.

Technical Session: Research

Light Armor Materials: H. P. George, Pitman-Dunn Laboratories, Frankford Arsenal.

Point Defects in Pure Nickel: J. J. Antal and Allen Goland, Materials Research Laboratory, Ordnance Materials Research Office.

The Role of Dilute Binary Transition Element Solutes and Time on the Recrystallization of Iron: E. P. Abrahamson II, Watertown Arsenal Laboratories.

Metallographic Investigation of a Multi-stage Piston Assembly Used in a Very High Pressure-High Temperature Apparatus: A. A. Giardini and J. E. Tydings, U.S. Army Signal Research and Development Laboratory.

Observation of Imperfections, Grains, and Surfaces

2:00 to 5:00 pm, East Ballroom

(Based on Submission of Research Abstracts)

Chairmen: N. Brown, University of Pennsylvania, and *J. P. Hirth, Carnegie Institute of Technology.*

Direct Observation of the Dislocation Structure in an Aluminum-Aluminum Oxide SAP-Type Alloy Using Transmission Electron Microscopy: G. S. Ansell, Rensselaer Polytechnic Institute, and Edwin Eichen, Allegheny Ludlum Steel Corp.

An investigation of the dislocation structure of an aluminum-aluminum oxide SAP-type alloy has been undertaken using the technique of transmission electron microscopy. The alloy used, Alcoa alloy M257, consists of a matrix of commercial purity aluminum containing a finely dispersed second phase of flake aluminum oxide particles. The dispersion spacing between these particles is approximately 0.4 μ. Starting from extruded rod of this alloy, a novel technique was employed to fabricate thin films suitable for use in transmission electron microscopy. Electron micrographs are shown which clearly picture representative dislocation structures found in this alloy. Two distinct types of dislocation arrays are seen throughout the matrix metal. The first which comprises the major portion of the alloy structure consists of an extremely fine network of intersecting stacking faults. The second type of structure, found only to a very limited extent in the material, consists of arrays of single or multiple dislocations either looped around the dispersed particles or piled up against them. These structures are interpreted in terms of dislocation models which have been proposed for both high-temperature creep (by Ansell and Weertman) based upon dislocation climb and the dislocation model proposed for yielding proposed by Ansell and Lenel. The dislocation structure observed is in good agreement with these two theories, particularly in connection with the large volume of material which consists of a very fine network of stacking faults in the aluminum matrix with only a small fraction of the alloy in which dislocation loops and arrays appear to be mobile. This lends support to the proposed reduction of active dislocation sources in these types of alloys as predicted by Ansell, and Ansell and Weertman.

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Transmission Electron Microscopy Studies on Ultramicrotomed Metals and Alloys: V. A. Phillips, General Electric Co.

An ultramicrotome employing a diamond knife was successfully used to cut a variety of metals and alloys, including alloys based on copper, nickel, lead, tin, aluminum, gold, silver and platinum. The structure of the slices obtained, which were about 100-600A in thickness, was studied by transmission electron microscopy. It seems clear that microtoming introduces high densities of dislocations, but recovery occurs rapidly at room temperature in low melting point materials such as lead, tin, and zinc rich phases, leading to sub-grain formation. In other materials, such as copper and silver, high densities of dislocations are frequently observed. Dislocations may be introduced by the cutting operation itself or by the bending which occurs as the slice is removed from the sample.

In certain grains in copper, silver and gold high densities of bands are observed running in one direction. These *fault bands* contain trains of widely dissociated glissile dislocations which may be on a single slip plane or on a group of parallel planes.

Very little macro-distortion of the structure is observed and second phase particles of sizes ranging from about 7 A to 1μ or more have been studied.

Radiation Hardening in Copper Single Crystals: I. G. Greenfield and H. G. F. Wilsdorf, Franklin Institute Laboratories

The mode of deformation of copper single crystals is considerably altered by irradiation with high energy neutrons of a dose of 10^{18} nvt (total flux). The general characteristics of the stress vs strain curve in fcc metals is observed; however, the curve is altered greatly by irradiation from what is found with non-irradiated high-purity copper. The important differences due to irradiation are: (1) the critical resolved shear stress is increased about eight fold, (2) the length of easy glide is increased, (3) serrations are noted during deformation in easy glide, and (4) the rate of hardening in the linear hardening region is decreased. In the past, the slip lines found in irradiated copper have been considered to be similar to those found in α-brass. In the present investigation, in which a high resolution electron microscopical technique was used, the slip line structure was found to be rather different. Instead of the deep steps common to alpha-brass, irradiated copper contained clusters of fine slip lines spaced about 90A apart.

The implications of the quantitative measurements of slip line parameters and of the direct observation of interactions of glide dislocations with radiation produced defects in thinned single crystals have been considered. The experimental results has led to an explanation of the mechanism of radiation hardening in copper.

Direct Observations on Twinning and Grain Growth in Austenite by Thermionic Emission Electron Microscopy: W. L. Grube and S. R. Rouze, General Motors Corp.

The processes of twinning and grain growth in austenite during annealing have studied utilizing thermionic emission electron microscopy. A series of plain carbon steels were employed for the studies which were conducted at temperatures in the range 850° to 1200°C. Results to date are in agreement with Burke's observations that, for fcc metals, grain boundary migration is necessary for the formation of annealing twins. Annealing twins in austenite may grow by extension in directions both perpendicular and parallel to the coherent twin boundary but only when the migrating twin boundary coincides with a grain boundary. Twin bands do not broaden by migration of the coherent boundaries but may broaden by the coalescence mechanism. These observations support the growth-accident hypothesis of twin formation suggested by Carpenter and Tamura and advanced by Burke through observations in α brass. Although the growth-accident mechanism may nucleate the twin, when a twinned region or a twin band is extended it is observed that it is not necessary that the generating grain boundary remain parallel to the composition plane. Contrary to the observations of Carpenter and Tamura on copper, annealing twins in austenite are thermally unstable and may be annihilated by either grain boundary or twin boundary migration. A twin band can be annihilated by twin boundary migration only by motion of stepped segments on the noncoherent boundary in a direction parallel to the octahedral twin boundary. The rate of migration of the noncoherent boundary segments in these cases varies over a wide range. Rates up to 190 μ per sec have been measured.

A motion picture film illustrating grain boundary migration and interaction, and twin formation and annihilation at the annealing temperature will be shown. The film also illustrates the particular advantage of cinematic recording in these studies where both time and event are recorded simultaneously as the action proceeds at temperature.

Cine Photomicrographic Studies of Mechanisms of Metal Whisker Growth: W. J. Allan and W. W. Webb, Union Carbide Metals Co.

Recent studies of the now well-known pocesses for growth of strong metal whiskers by reduction or decomposition of volatile metal halides have revealed that the mechanisms are quite different from those first anticipated. Ciné photomicrography at magnifications up to 100X and camera speeds ranging from ½ to 64 frames per sec have provided a means of observing the mechanisms of these growth processes. Growth rates (as a function of whisker length) have been measured for the several reactions and the results compared with various theories and models of the growth process. Growth rates as high as 0.1 mm per sec have been observed to produce whiskers of nearly theoretical strength. Except in the growth of palladium whiskers by thermal decomposition of $PdCl_2$ the growth rates are independent of whisker length. It has been found that the reduction reaction takes place in a condensed phase on the growing surface in most cases. Many of the peculiarities of growth form that have been observed and previously reported can be understood from these results. For example, the formation of kinked and branched whiskers, irregular filaments, helices, etc., can be seen to be a natural consequence of the growth mechanisms. The observations suggest some interesting implications about the mechanisms of some of the high-temperature reactions involved in metallurgical processes. Motion pictures of the growth of palladium copper and silver whiskers and other crystals will be shown to demonstrate the mechanisms discussed.

Dislocations in Zinc: V. V. Damiano and G. S. Tint, Franklin Institute Laboratories.

A technique of cinephotomicrography and continuous etching has been developed to reveal on 16mm motion picture film the three dimensional aspects of dislocations and dislocation networks in zinc crystals. Lengths of dislocation lines were revealed by etching a cleaved (0001) surface after room temperature aging of crystals containing 0.030 pct Cd. The continuous dissolution of the (0001) surface reveals long continuous slip dislocations, tilt boundaries and hexagonal networks. The hexagonal networks are seen to lie on the (0001) and were produced by annealing a deformed crystal for several hours at 200°C. The results suggest that slip dislocations with different Burgers vectors interact to form the networks. The dislocations in the hexagonal nets were seen to be oriented in the screw orientation. Sessile dislocations which emerge at the (0001) surface were observed as single etch pits. These were seen to be located frequently at the nodes of the hexagonal networks. The technique enables one to reconstruct three dimensional models of dislocation networks.

This work has been sponsored by the OSR of the United States Air Force.

Dislocation Etch Pits on the (0001) Surface of Zinc Crystals: H. S. Rosenbaum and M. M. Saffren, General Electric Co.

An etch pit technique has been developed for revealing dislocations which intersect the basal plane of zinc. The etchant involves either a halogen (Br or Cl_2) or a halogen acid (HI, HBr, or HCl) dissolved in a polar organic solvent such as alcohol, ether or glycerol. The pits produced by HI, HBr, or Br in such a solvent are hexagonal pyramids with the sides of the hexagon parallel to the close-packed $<11\bar{2}0>$ directions in the basal plane. The pits produced by HCl or Cl_2 are either conical or hexagonal pyramids; in this case, the sides of the hexagon are parallel to $<10\bar{1}0>$ directions. In all cases the pits are produced with no apparent decoration required.

The following evidence is offered to show that the pits correspond to dislocations which intersect the basal plane surface.

1. The etch pit patterns on the two halves of a cleaved crystal match each other.
2. Deformation of the crystal causes non-basal glide which is seen as bands of etch pits parallel to $<10\bar{1}0>$ directions on the basal plane.
3. Annealing the crystal after deformation causes a redistribution of the etch pit pattern.

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4. Flat-bottomed pits are produced when, after an initial etch, a stress moves the dislocations to new positions and the crystal is again etched.

The etching technique has been used to observe slip bands, twins and kinks.

Slip Bands: Non-basal slip is seen to occur readily at room temperature or in liquid N_2. By correlating the etch pit bands on the basal plane with deformation markings on a polished surface perpendicular to the basal plane, the second order pyramidal slip plane {1122} was confirmed. On annealing, the dislocations rearrange themselves in such a way that they lie in the basal plane leaving kinks on the basal surface with few etch pits along the kinks.

Twins: A row of dislocations is seen to emanate from the tip of mechanical twins. These dislocations apparently lie on the twinning plane.

Kinks: One can distinguish kinks, which are manifestations of dislocations lying parallel to the basal plane, from other markings like slip bands which are composed of dislocations intersecting the basal plane.

Surface Markings and Their Relation to Dislocations in Aluminum: J. M. Lommel, General Electric Co.

When a smooth-surfaced sample of aluminum is cooled from a high temperature, pits develop on the surface. Dougherty and Davis have shown that these pits are formed by the condensation of vacancies from a lattice which has been supersaturated. The supersaturation arises from the decrease in the equilibrium concentration of vacancies with decreasing temperature, and occurs in regions removed from such vacancy sinks as grain boundaries, subgrain boundaries, and previously formed pits. It has been suggested that pits nucleate at sites where dislocations intersect the surface.

X-ray diffraction microscopy has been used to compare the distribution of dislocations with the distribution of vacancy pits formed on heat-treated samples. Since the X-ray technique cannot be used on heavily oxidized samples, the comparison has been made after several different treatments of the surface. Electropolishing and ion bombardment have been used to clean the surface of pitted samples. Although pit and dislocation densities are nearly the same in the region investigated, no one-to-one correspondence between pit sites and dislocations has been observed. Dislocations are found which have not nucleated vacancy pits in regions free from subboundaries.

A comparison has been made between dislocations and pits formed with the solutions devised by Wyon, Marchin and Lacombe. No correlation was observed between dislocations revealed by X-ray diffraction microscopy and the large geometric pits which can be formed in aluminum. By suitable choice of the etchant composition small pits, called microfigures, can be developed. Subgrain boundaries delineated by these pits are the same as those observed in X-ray micrographs. A correlation has been found between these pits and decorated dislocations.

Etch-pit Characteristics of Molybdenum: M. A. Adams and A. Iannucci, Materials Research Corp.

In the course of an investigation of sub-structure formation in molybdenum a study was made of the etch-pitting produced by—

(a) chemical attack in a 1:1 mixture of 30 pct aqueous potassium ferricyanide and 10 pct aqueous sodium hydroxide.
(b) electro-chemical attack in 10 pct aqueous sodium hydroxide.
(c) electro-chemical attack in ½ pct aqueous oxalic acid.

Initial experiments with single crystals containing prominent crystallographic surfaces indicated that tech-pits are formed on {100} but not on {111} or {110} planes, and this result was confirmed by studies on spherical single crystals which showed that all three etching treatments cause pitting only on those surfaces which have poles within 28° ± 1° of <100>.

Examinations of etched matching cleavage faces of fractured single crystals revealed, at least in those areas remote from cleavage steps, a one to one correspondence of many of the etch-pits on the two faces as would be expected if the pits nucleate on dislocations.

When a given surface is subjected successively to each of the three etching treatments it is found that treatments (a) and (b) produce pits on exactly similar sites but that treament (c), as well as causing attack on these sites, produces pits in roughly equal number of new sites. From this it appears that chemical etching in a potassium ferricyanide-sodium hydoxide mixture, and electro-etching in sodium hydroxide, may cause pitting on the sites of dislocations of one type (edge or screw), whilst electro-etching in oxalic acid may cause pitting at the sites of both types of dislocations. These results indicate the care that must be taken in interpreting the results of etch-pit examination on molybdenum.

This work was sponsored by the Materials Laboratory, Wright Air Development Center.

Primary Oxide Films Formed on Molten Metals and Alloys. Electron Diffraction Study: Goro Shimaoka, National Research Institute for Metals. (Japan).

Using newly devised specimen-adaptor for study of surfaces of liquid metals, the freshly prepared surfaces of molten tin, lead, bismuth, lead-tin, lead-zinc, lead-cadmium and lead-antimony alloys in the vacuum ($\sim 10^{-4}$ mmHg) at the temperatures little above their melting points to 500°C were observed by electron diffraction reflection method. It was found that the surfaces of these metals and alloys were covered with very thin oxide layers ($\sim$10Å thickness). These primary oxide films consisted of small crystallites oriented preferentially with a lattice-plane characteristic of each crystal (in general, with the most densely packed plane of atoms) parallel to the metal surface. On the molten tin, SnO crystals were oriented with the (001) plane at relatively low temperatures and SnO_2 crystals with the (101) planes at higher temperatures; on the molten lead, orthorhombic PbO crystals with the (001) plane; on the molten bismuth, tetragonal β-Bi_2O_3 crystals with the (221) plane; on the molten Pb-65 pct Sn and Pb-35 pct Sn alloys, SnO crystals with the (001) plane and SnO_2 crystals with the (101) plane; on the molten Pb-1 pct Sn alloys, SnO_2 crystals with the (101) plane; on the molten Pb-0.5 pct Zn alloy, ZnO crystals with the (0001) plane; on the molten Pb-1pct Cd alloy, CdO crystals with the (001) plane; and on the molten Pb-1 pct Sb alloys, Sb_2O_3 crystals with the (111)$\sim$(001) plane parallel to the metal surface. These orientation of the oxide crystals were found to be essentially the same as those obtained with the oxide films formed in air.

On A New Technique for Studying Oxide Films on Aluminum: P. E. Doherty, P. C. Johnson, and R. S. Davis, Arthur D. Little, Inc.

Small surface pits are observed to form on electropolished surfaces of aluminum single crystals during cooling from elevated temperatures. These pits are attributed to the condensation of vacancies from a supersaturated solution. The pits form under the Al_2O_3 layer. The oxide over the pits may be stripped off with a citrocellulose replica. The oxide particles adhere to the replica and may be examined directly by electron microscopy. Very thin oxides may be examined by this technique. Oxide thickness, grain size, diffraction patterns, and general appearance have been examined. Single crystal orientation and oxidizing conditions have been varied.

Recent Advances in the Metallurgy of Stainless Steels

2:00 to 5:00 pm, Pennsylvania Room-East

(Sponsored by IMD Corrosion Resistant Metals Committee)

Chairmen: S. A. Rosenberg, National Bureau of Standards, and L. R. Scharfstein, The Carpenter Steel Co.

Effect of Composition on Stress Corrosion Cracking of Stainless Steels: D. van Rooyen, Westinghouse Electric Corp.

Effect of Zerolling on the Mechanical Properties of Austenitic Stainless Steels: J. R. Mihalisin and D. W. McDowell, International Nickel Co.

It has been shown that the *metastable* austenitic stainless steels can be strengthened by plastically deforming them at sub-zero temperatures and thus promoting the γ to α transformation. On the basis of equal ductility, it was found that the zerolled stainless steel exhibited higher strengths than the same steels deformed at room temperature.

Reductions of 70-80 pct at −105°F develop strength properties in austenitic stainless steels beyond the limit obtainable by cold rolling at room temperature regardless of the amount of reduction. With reductions of 60 pct or less, strength increased with decreasing rolling temperature from 75°F to −320°F, the greater increase occurring between 75°F and −105°F. Further reductions in the same temperature ranges result in further increases in strength level; however, the increase is proportionately less.

It has been established that stress relieving treatments following the subzero rolling can result in still further increases in strength.

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A combined rolling treatment (warm + subzero or vice versa) show that the sequence of rolling is important.

Elevated temperature strength can be retained at a higher temperature by increasing the amount of cold reduction rather than lowering the temperature of reduction. This is true regardless of the strain rate and extending down to room temperature tests.

Temperature Dependent Fatigue and Damping in 12 Cr Steels with Delta Ferrite: V. K. Chandhok and E. J. Dulis, Crucible Steel Corp.

Effect of Boron on Hot Ductility and Carbide Precipitation in Austenitic Stainless Steels: F. K. Bloom and E. E. Denhard, Armco Steel Corp.

High temperature tensile samples of boron-treated and non-treated 25 pct Cr-20 pct Ni steel were fractured at varying strain rates at temperatures ranging from 1700°F to 2300°F. The results showed that boron markedly increased hot ductility.

In general, ductility increased with strain rate and temperature for both materials. At the lowest test temperature, and independent of strain rate, little difference was noted in the fracture ductility of boron treated and non-treated steel. The greatest difference was noted at 2000°F at intermediate strain rates, although this observation should be qualified because of the deboronization that occurred in the treated samples at the lower strain rates and higher temperatures. At those temperatures, where carbides were out of solution, straining was accommodated by elongation of the primary austenite grains. At higher temperatures, grains fragmented, or developed sub-grains which continued to grow until fracture ultimately occurred. Non-treated steel, dependent on strain rate and temperature, developed grain boundary voids which lead to premature brittle failure.

Grains that fragmented during the straining process remained equiaxed to the point of fracture, despite the fact that they elongated as much as 95 pct.

Current theories advanced to explain the improved ductility in stress rupture of boron-treated steels are not applicable to the present investigation. The appearance of primary grain boundary fractures suggests that void formation by vacancy condensation might be a possible mechanism for elevated temperature failure. This fits the observation of increased ductility with increasing strain rate, but does not explain why the ductility increased steadily with temperature.

Effects of Delta Ferrite on Hot Cracking Tendencies of Stainless Steels: F. C. Hull, Westinghouse Electric Corp.

Ingot Structure Control II

2:00 to 5:00 pm, Pennsylvania Room-West

(Sponsored by IMD Melting and Casting Committee)

Chairmen: W. A. Tiller Westinghouse Electric Corp., and H. W. Lownie, Jr., Battelle Memorial Institute.

Relationship Between Small Scale Experiments and Full Scale Production: B. Chalmers, Harvard University.

Unidirectional Solidification of Steel Ingots: M. C. Flemings, S. Z. Uram, R. V. Barone, and H. F. Taylor, Massachusetts Institute of Technology

A summary is presented of work conducted at M.I.T. over the last two years relating to solidification of high-strength low-alloy steel ingots. Ingots weighing approximately 50 lb were cast using techniques to achieve varying degrees of directional solidification. Optimum directionality (essentially unidirectional heat flow during solidification) was obtained by employing a mold with a water cooled base plate; sides and top of the mold were insulated with moldable exothermic material. By this technique, ingots were cast with fully columnar macrostructures; columnar grains grew perpendicular to the water cooled base plate and extended to the top of the ingot. Rate of freezing was proportional to the square root of time.

Careful measurements were made of microporosity, dendritic structure and segregation in the experimental ingots; results of these measurements are interpreted. Micro-porosity (determined by microradiographic techniques) is essentially absent in unidirectionally solidified ingots, except at the topmost portions. For comparison, microporosity was found to be the order of 1 pct in ingots made by more conventional techniques. Dendrite arm spacing increases linearly with distance from the chill. Unidirectionally solidified ingots are remarkably free of macrosegregation, the only significant segregation being an increase in sulfur and silicon at the top of the ingots due to inclusion flotation during freezing.

Mechanical properties of unidirectionally solidified ingots are presented; ductility is shown to exceed greatly that of ingots made by conventional techniques. For example, in one ingot (in the heat treated condition) mechanical properties parallel to the columnar grains averaged approximately 290,000 psi UTS, 230,000 psi yield strength, and 17 pct reduction in area; properties were essentially the same transverse to the columnar grains. In a comparable ingot produced in a sand mold average properties obtained were respectively 275,000, 225,000, 6 pct. The improvement in properties obtained by unidirectional solidification is attributed to (1) reduction of micro-porosity, (2) improved chemical homogeneity on a micro and macro-scale, and (3) increased freedom from inclusions. As a result of improved soundness and homogeneity, unidirectionally solidified ingots should prove to be more readily forgeable than conventional ingots; also, wrought material produced from such ingots should be of unusually high quality (i.e., possess high transverse ductility, little or no chemical or other heterogeneities, etc.)

Useful and Practical Mathematical Description of Solidification: E. L. Foster, B. Fletcher, and C. K. Franklin, Battelle Memorial Institute

The heat flow from a solidifying right cylindrical casting was described in a mathematical model suitable for solution by high-speed digital computer equipment. The model was constructed by dividing the casting and mold into a number of cells of a size and shape which are adequate to develop the basic equations of heat flow. Temperatures for these cells are calculated at short time intervals from the time of pouring. Such casting variables as heat flow from casting ends, pouring time, separation of the casting from the mold (when heat transfer changes from conductive to radiative), temperature dependence of thermal properties of liquid and solid metal and mold materials, emissivities of the various radiating surfaces, and the heat of fusion can be evaluated with the model.

In an experimental program conducted concurrently with the development of the analytical methods, computed predictions of the time-temperature history of the individual cells, solidification patterns in the ingot, and changes in thermal gradients during solidification proved accurate in the vacuum-induction casting of uranium. While the symmetry of the ingot and mold, the solidification of uranium at a constant temperature, and the absence of a heat-transporting gaseous atmosphere simplified the construction of the model, the general validity of the theoretical approach for more complex systems seems indicated.

Diffusion

2:00 to 5:00 pm, Hall of Flags

(Based on Submission of Research Abstracts)

Chairmen: C. A. Wert, University of Illinois, and J. N. Hobstetter, University of Pennsylvania

Quantum Effects on the Diffusion of Hydrogen in Metals: W. R. Heller, International Business Machines Corp.

Experiments on diffusion and solubility of hydrogen in metals are summarized, with particular emphasis on metals not known to form hydrides. An explanation is proposed for the rather limited range of values of the heat of solution in such metals. Correlation of D_o and ΔH values from diffusion measurements is found to suggest that quantum tunneling of dissolved hydrogen atoms from one interstitial position to another reduces both D_o and ΔH. Experiments of the author, measuring what appears to be a Snoek-type internal friction peak due to hydrogen in iron, are cited to demonstrate the quantum effects upon diffusion in another way.

Solubility and Diffusivity of Hydrogen in Molybdenum: M. L. Hill, John Hopkins University.

The diffusivity of hydrogen in molybdenum was determined from measurements of the non-steady rate of evolution of hydrogen from specimens that had been heated in hydrogen at 1 atm pressure

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at temperatures from 1250° to 1700°C and then quenched in water. The diffusivity conformed to the relation

$$D_{\text{cm}^2\text{ per sec}} = .059\, e^{-14,700/RT}$$

over the entire range of temperatures studied, which extended from 575° to 980°C.

Based on these diffusion coefficients, it is concluded that no significant quantity of hydrogen was lost during the few seconds required to cool the specimen from the charging temperature. Therefore the solubility at the charging temperature could be obtained from measurements of the total hydrogen content of the quenched samples. Such measurements were made as a routine part of the evolution rate measurements. The solubility values obtained in this way represent a considerable extension in the range of temperatures over which previous studies have been conducted. The present results tie in quite well with those obtained by Sieverts at from 600°C to 1100°C to yield the following relation between solubility and temperature.

$$S_{\text{cm}^3\text{/100 g}} = 27\, e^{-9700/RT}$$

These results clearly show that the solubility continues to increase with increasing temperature up to 1700°C and it therefore appears that the anomaly reported by Martin, wherein the solubility reached a maximum at 800°C, was an artifact.

The present results have practical value in commercial processing of molybdenum since they not only indicate the degree of hydrogen contamination that will occur during sintering or annealing in hydrogen but also show that suitable heat treatment can be devised for the removal of hydrogen.

Diffusion in a Ferromagnetic Alloy: James Stanley and C. A. Wert, University of Illinois.

Diffusion constants in an alloy of Fe + 18 pct V have been determined over a wide temperature range by a combination of radioactive tracer and anelastic methods. The region of measurement extended over a considerable region on both sides of the Curie temperature. In the paramagnetic region the diffusion coefficients of iron and vanadium as a function of temperature obey the relations:

$$D(Fe^{59}) = 7\, e^{-61,700/RT}$$
$$D(V^{48}) = 4\, e^{-59,500/RT}$$

Below the Curie temperature the anelastic data show that diffusion is much slower than these two expressions indicate; it is not possible to determine beyond question which of the two diffusion coefficients is changed the most. Diffusion in the well ordered ferromagnetic state is about 100 times less than would be expected from either of the above equations. Part of this effect appears to be a change in activation energy and part a change in D_o. The interpretation of the effect is not straightforward because theories of the activation energy and of D_o do not allow accurate enough calculations to be made from the known properties of the alloy. Part of the change in D_o may be due to a change in the shear modular observed near the Curie temperature.

This research was supported in part by the Air Force Office of Scientific Research.

Self-Diffusion of Iron and Sulfur in Ferrous Sulfide: R. H. Condit, University of California, and C. E. Birchenall, University of Delaware.

Measurements of the self-diffusion or iron and sulfur in ferrous-sulfide single crystals are reported as a function of temperature, deviation from stoichiometric composition, and crystallographic direction. For the temperature region, 250°-697°C, the diffusion coefficient for iron is

$$D = D_{o_{\perp}}\,(d^2 + 10^{-3})^{\frac{1}{2}} \exp(-4/RT)\,(d^2 + 10^{-3})^{\frac{1}{2}} \exp(-(19 + 20d)/RT)$$

where d is the iron deficit as used in the formula, $Fe_{1-d}S$, $D_{o_{\parallel}} = 0.16$ sq cm sec^{-1} and $D_{o_{\perp}} = 0.27$ sq cm sec^{-1}, the frequency factors for diffusion in the direction normal to and parallel with the c-axis of the hexagonal crystal, respectively, and RT has its usual meaning and is expressed in units of kcal per atom. Below the Néel temperature, 330°C, the diffusion coefficient appears to decrease with increasing iron deficit as would be expected on the basis of previous evidence for ordering of vacancies on iron sites. Rates of sulfur diffusion in the temperature range below 700°C are more than 10^6 smaller than for iron. The results of this investigation are in good agreement with measurements by Meussner and Birchenall (1957) of the rates of sulfidation of iron in sulfur atmospheres.

Iron self-diffusion is primarily by a vacancy mechanism, the vacancies being provided by the iron deficit plus some intrinsic disorder. Details of the diffusion mechanism are discussed.

This investigation was carried out at Princeton University under contract with the Air Force Office of Scientific Research, ARDC.

On the Internal Friction of Cold Worked and Quenched (Martensitic) Iron and Steel: I. Tamura, Osaka University, and T. Mura and J. O. Brittain, Northwestern University.

The internal friction peaks which are observed at about 240°C for cold-worked iron and steel and about 260°C for steels in the martensitic condition are explained as due to growth and solution of a carbide precipitate as a consequence of an oscillating dislocation. The activation energies associated with the peaks were about 38,000 and 40,000 cal per mol respectively.

The theory for the peaks is based upon the addition of a term to the free energy in order to account for the strain energy due to the interaction of the solute element and the line imperfections. The linear standard solid was obtained from a model in which dislocations are vibrating with an atmosphere of carbide precipitates. The agreement between theory and observation seems reasonable.

This research was supported by the U. S. Air force through the Air Force Office of Scientific Research of the Air Research and Development Command.

Diffusion in Liquid Amalgams: P. C. Mangelsdorf, Jr., University of Chicago.

The diffusion coefficients of a number of dilute metallic solutes in liquid mercury at room temperature have been determined. The results are compared with several current theories of diffusion in liquids.

This work was supported in part by the U. S. Office of Naval Research.

Internal Friction in Liquid Metals: Gerald Abowitz, Cornell University, and R. B. Gordon, Yale University.

In a liquid internal friction is expected to arise through shear viscosity and thermal conductivity. The magnitude of these losses can be calculated using equations due to Stokes and Kirchoff. Any *excess* damping over and above that due to these causes has its origin in atomic or molecular processes occurring in the fluid.

The attenuation of sound in a series of liquid Hg-Tl alloys has been measured by the ultrasonic pulse technique over a range of temperatures and frequencies. In pure mercury the observed damping is very nearly the same as that calculated as being due to the shear viscosity and the thermal conductivity. The calculated and observed temperature coefficients of the damping also agree well. With increasing thallium content there is an increasing excess attenuation. Also, as more thallium is added the temperature coefficient of the attenuation decreases, passes through zero, and then becomes negative. Since the shear components of stress in the longitudinal ultrasonic wave are fully relaxed, the excess attenuation is attributed to a volume viscosity associated with the dilatational component of the longitudinal wave. Interpreted in terms of a relaxational process, values are obtained for τ_v and Q, the activation energy, suggestive of a process involving individual atomic movements as responsible for the volume viscosity. It is suggested that the process may be similar to that of the Zener relaxation in solid solutions.

Self Diffusion of Germanium in Pure and Doped Crystals: H. Widmer, G. R. Gunther-Mohr, and A. S. Nowick, International Business Machines Corp.

Self diffusion of Ge^{71} isotope was studied using two techniques, a residual activity technique and a surface activity method. The former technique, which is based on the measurement of the residual activity after removal of thin sections, is capable of measuring diffusion coefficients down to 10^{-15} sq cm per sec (penetration depth ~ 1 μ) to a precision of better than 10 pct. The results obtained for the self diffusion of Ge^{71} in pure crystals give an activation energy Q = 69.4 ± 0.4 kcal per mole and D_o = 10.8 sq cm per sec, in reasonable agreement with earlier measurements.

The diffusion coefficient of Ge^{71} is drastically affected by the presence of small amounts of electrically active impurities. Thus, for example, the presence of 0.2 mole pct of gallium in germanium

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depresses the self diffusion coefficient to 0.4 of its value in the pure crystal. These results are interpretable in terms of the effect of shifts in the Fermi level in producing changes in the concentration of vacancies.

Thermal Diffusion in Non-stoichiometric Zirconium Dihydride: A. W. Sommer and W. F. Dennison, Atomics International

Measurements on the magnitude and direction of the Ludwig-Soret effect in Δ phase ZrH_x in the temperature range 1000°-1300°F have been made. The *heat of transport*, Q^*, of hydrogen in ZrH_x was determined to be 1.3 ± 0.4 kcal per mole. A mathematical analysis of the kinetics of hydrogen redistribution is presented and shown to compare favorably with experimentally observed results. A similarity in the magnitude of Q^* and H, enthalpy of solution, was noted. Instances of this similarity, as found in other works, are reported. A rationalization is presented to account for the similarity between Q^* and H.

This work was sponsored by the Aircraft Reactors Branch, Division of Reactor Development, AEC.

Thermodynamics of Diffusion at High Pressures: R. W. Keyes, International Business Machines Corp.

The way in which the strain energy model of an atomic defect can be used to predict the effect of pressure on diffusion rates will be explained in this paper. The rate of a diffusive process in a solid is controlled by the isobaric, isothermal work which must be performed to create the atomic defect necessary for the diffusive process. This work is the Gibbs free energy of activation, ΔG^*. The pressure derivative of ΔG^* is the volume of activation, ΔV^*. A measurement of the pressure dependence of a rate of diffusion determines the parameter ΔV^*.

On the strain energy model ΔG^*, the work which is performed in creating an atomic defect, is regarded as ordinary elastic work, and can be discussed with the aid of the usual thermodynamic theory of elasticity. The thermodynamic theory shows that the dilation associated with elastic energy is determined by the pressure derivative of the elastic constants. When the dilation is identified with ΔV^* the result is

$$\frac{\Delta V^*}{\Delta G^*} = \left(\frac{\delta \ln \mu}{\delta P}\right)_T - \chi$$

where μ is an elastic modulus and χ is the compressibility. Introduction of the Grüneisen model to describe the pressure variation of the elastic modulus reduces Eq. (1) to

$$\Delta V^* = 2(\gamma_G - 1/3)\chi\Delta G^*$$

A comparison of Eq. (2) with available experimental data shows that the predicted proportionality is actually present. However, the observed ΔV^* are generally about 40 pct larger than the calculated values. If this extra factor of 1.4 is introduced into Eq. (2) a quite accurate relationship for the prediction of activation volumes results.

Effect of Hydrostatic Pressure on Self-diffusion in Lead: J. B. Hudson and R. E. Hoffman, General Electric Co.

The self-diffusion coefficient of pure lead has been measured at five pressures between atmospheric and 40 kb, over a temperature range of about 150°C near the melting point at each pressure. Measurements were made using a radioactive tracer technique. Samples were sectioned electrochemically after the diffusion anneal to determine the diffusion penetration curve. The pressure was applied in a *belt* type pressure device similar to that used by Hall *et al* in the diamond synthesis.

The sectioning technique developed was capable of removing sections less than 1 μ in thickness, and thus permits measurement of diffusion coefficients as low as 3×10^{-14} sq cm per sec. Comparison of results obtained using this technique at atmospheric pressure with previous studies using conventional sectioning techniques shows excellent agreement.

The self-diffusion coefficient, D, can be represented by an equation of the form

$$D = D_o e^{\frac{-\Delta H^*}{RT}}$$

at any pressure, where ΔH^*, the activation energy is constant with temperature. At constant temperature D is given by an equation of the form

$$D = D_o' e^{\frac{-P\Delta V^*}{RT}}$$

where ΔV^*, the activation volume, appears to be constant with pressure at least up to 20 kb. Above this pressure ΔV^* appears to decrease with increasing pressure. An apparent correlation between the effects of pressure on self-diffusion and on the melting point of the material observed by Nachtrieb and co-workers at pressures below 10kb was found to be only approximate as measurements are carried to higher pressures.

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Titanium

9:00 am to 10:30 am, West Ballroom

(Based on Submission of Research Abstracts)

Chairmen: D. J. McPherson, Armour Research Foundation, and *G. E. Spangler, Franklin Institute Laboratories.*

Phase Transformations During the Heat Treatment of the Alloy Ti-6Al-4V: P. J. Fopiano, Manufacturing Laboratories, Inc., and M. B. Bever and B. L. Averbach, Massachusetts Institute of Technology.

The effects of solution and aging treatments on the alloy Ti-6Al-4V were investigated by hardness measurements, light and electron microscopy, resistance measurements at the aging temperature, and X-ray diffraction. For one selected solution treatment, the effect of aging was also investigated by measurements of; the resistance at room temperature, the expansion coefficient, the precision length and the specific volume.

The property changes resulting from the solution and aging treatments are related to the equilibrium alloy content of the phases present after the solution treatment. The vanadium content of the β phase and the aluminum content of the α phase are especially important. The equilibrium vanadium content of the β phase as estimated from the published phase diagram was confirmed for the solution-treatment temperatures and extrapolated to the conventional aging temperatures for the alloy Ti-Al-4V.

The hardness has two maxima as a function of aging time after solution treating in the $\alpha + \beta$ region. The first maximum is associated with the decomposition of martensitic α and has similar kinetic features as the decomposition of martensite in the Ti-V system. The second maximum is attributed to the precipitation of an ordered phase, which is shown to be diffusion controlled.

Support of this research by the Office of Naval Research under Contract Nonr-1841(02) is acknowledged.

Grain Boundary Segregation of Carbon, Nitrogen, Oxygen in Titanium: Joseph Winter, Olin Mathieson Chemical Corp., and Irving Cadoff, New York University.

The partitioning or segregation of excess interstitial solutes at the grain boundaries in α phase and β phase titanium alloys was investigated. Internal friction spectra of dilute alloys of titanium with oxygen, nitrogen and carbon were obtained. From these

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spectra the activation energy for grain boundary stress relaxation, the net interaction energy of a solute atom with a grain boundary, and the excess solute concentration were calculated.

As in previous investigations of this type the partitioning of excess solute was found to obey a relation of the form

$$C_s = C_o \exp V_o/kT$$

The interaction energies were found to lie in the range 0.15 to 0.40 ev for binary interstitial alloys. The expected behavior of increase in activation energy, decrease in peak height and decrease in peak temperature with increasng solute concentration was observed. In the case of carbon and nitrogen alloys the activation energy increase exhibited a saturation effect. While this was not observed in oxygen alloys, the nature of the curve indicated approach to saturation. In the range of saturation, the carbon alloys developed a second peak, the composition at which the peak was fully evident being close to the solubility limit of carbon in α titanium. From the nature of the curves obtained it may be inferred that the double peak may be associated with the formation of the Δ phase, which causes local depletion of the carbon atoms segregated at the grain boundary.

Tensile measurements indicated that the increase of excess solute at grain boundaries and dislocations brought about by either increasing the bulk concentration or decreasing the equilibrium annealing temperature resulted in an increase in yield strength. The effect of excess solute on yield stress was found to be parallel to its effect on the internal friction spectra.

This research was supported by the United States Air Force under Contract No. AF33(616)-5506 monitored by the Materials Research Laboratory, Wright Air Development Center.

Properties of Zone Refined Titanium: G. E. Spangler and Marvin Herman, Franklin Institute Laboratories.

The recrystallization characteristics and grain growth kinetics of zone refined titanium have been determined. Both the recrystallized grain size and the rate of grain growth are strongly impurity dependent, as evidenced by a 10-fold decrease in recrystallized mean grain diameter and a progressive increase in activation energy for grain growth from the pure to impure end of a zone refined bar. The activation energy for grain growth was found to be 38,000 cal per g-atom for the purest portion of the zone refined material. The grain growth kinetics for all portions of the zone refined bar obeyed a $D^2 - D_o^2 = Kte^{-Q/RT}$ relation.

The tensile properties of the zone refined titanium have been determined from room temperature to 4.2°K. The proportional limit of this material at a 0.05mm grain size is 3000 psi at room temperature with about 60 pct elongation. While the maximum rate of grain growth was observed near the starting end of the zone passes, the minimum tensile strengths were observed near the finishing end. This apparent difference in response to impurity concentration can be qualitatively explained on the basis of the difference in redistribution characteristics of the interstitial impurities and metallic impurities in titanium. Observations are also presented and discussed on the effect of purity and grain size on the tendency for discontinuous plastic flow at 4.2°K.

This work has been sponsored by the OSR of the United States Air Force.

Deformation Textures of Titanium Alloys: P. A. Albert, Westinghouse Electric Co., Y. C. Liu, Ford Motor Co., and I. Cadoff, New York University.

Wire and rolling textures in beta titanium alloys of stable β, α plus stable β, metastable β, α plus metastable β, and α plus α' structures were investigated.

Drawn wires of stable beta alloys were found to have the <100> direction parallel to the fiber axis, and the rolling texture observed for the stable β was a combination of (100) <100> and (112) <100> components of approximately equal intensity.

Institute of Metals Lecture

11:00 am, West Ballroom
C. C. Long and J. H. Jackson, Presiding

The Work Hardening of Metal Crystals—to be presented by Professor N. F. Mott, F.R.S., University of Cambridge, England.

Phase Transformations

9:00 to 10:30 am, East Ballroom

(Based on Submission of Research Abstracts)

Chairmen: H. M. Otte, RIAS, and W. Liebmann, Radio Corporation of America.

The (225) Martensite Transformation in Iron Alloys: C. M. Wayman and T. A. Read, University of Illinois, and J. E. Hanafee, International Nickel Co.

Phenomenological theories on the formation of martensite have been developed. But in the case of alloys of iron where plate-like martensite is formed, owing to the difficulties in obtaining experimental crystallographic information, there is insufficient data to test the theories.

For an accurate study of the transformation it is necessary that the martensite habit plane and orientation relationship for a single plate be determined. These observations are especially needed for the $(225)_A$ transformation for which this determination had not previously been made. Such an analysis is reported here for an alloy of Fe-7.90 wt pct Cr-1.11 wt pct C for which a habit plane near $(449)_A$ was determined. The theories of Wechsler, Lieberman and Read (WLR) and Bowles and Mackenzie (BM) were examined in the light of the experimental results of this investigation. These theories although equivalent differ in the interpretation of the $(225)_A$ transformation. The habit plane and orientation relationship predicted by both theories were in good agreement with the experimental measurements. With each theory, the predicted habit plane normal came within 3 degrees of the experimental one. By working the theories in reverse, the inhomogeneous shear of the transformation was determined. The experimental results agree with the predictions of the WLR theory, provided that the inhomogeneous or lattice invariant shear is assumed on the (011) plane of the martensite. The BM theory is equally good for a martensite inhomogeneity on (112) assuming that the total distortion includes on isotropic dilation. In each case the shear direction is the close-packed $<111>_M$ direction. Some microscopic observations on the subject alloy indicated that the interface plane may not be one of zero average distortion, as supposed by WLR, and that a dilation in the interface appears reasonable. In the work described herein the presence of martensite sideplates was observed. The nature of these plates is uncertain at present. From this investigation, the findings point to the need for a microscopic examination of the austenite-martensite interface. Also, further study should be made of the interface dilation parameter, possibly in terms of an array of transformation dislocations which generate the new lattice from the old.

Study of the Martensitic Transformation in Iron-base Alloy Single Crystals: J. F. Breedis and W. D. Robertson, Yale University.

The influence of applied stress and temperature on the transformation has been studied in single crystals of iron—16 pct Cr—12 pct Ni adn iron—30 pct Ni alloys. As found previously, a hcp (ϵ) phase formed together with the bcc martensite on cooling the Cr-Ni alloy. A two surface analysis revealed the apparent habit plane in Fe-16Cr-12Ni to be $(111)_A$; the orientation relationship determined may be described as:

$$(111)_A \parallel (00.1)_\epsilon \parallel (101)_M$$
$$[110]_A \parallel [12.0]_\epsilon \parallel [111]_M$$

The relationship between the fcc and bcc phases is approximately that of Kurdjumov and Sachs.

The critical resolved shear stress of the iron—30 pct Ni alloy (habit ~ (3, 10, 15)$_A$) increased steeply from 10.3 kg per sq mm at 325° to 73.3 kg per sq mm at −12°C as the m_s temperature (−21°C) was approached. At −12°C, only four to five of the possible martensite habits were produced. In the Cr-Ni alloy, in which the habit is also the octahedral slip plane, a significant increase in the shear stress occurs between 200°C and 100°C (as compared with a more stable Fe-16Cr-25Ni alloy (m_s<−197°C) which showed the usual variation of CRSS with temperature). Below 100°C, the apparent CRSS, calculated by assuming a resolution of shear stress on the most favorably oriented plane and in the favorable shear direction, decreased rapidly to zero at the m_s temperature (−32°C). Further, decreasing the temperature while maintaining a constant elastic stress (based upon the temperature dependence of the Fe-16Cr-25Ni alloy) completely restricted the transformation to that plane having the highest shear stress.

The effects of prior deformation upon subsequent transformation

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and vice versa, and the effectiveness of grain and twin boundaries and slip bands as barriers to martensite formation were also studied.

Examination of the reversion of the transformation showed that the hcp phase disappeared below 400°C, and the bcc phase mostly between 500°C and 600°C. After reversion to austenite, martensite was observed to reform at the same sites that had transformed initially, thus demonstrating crystallographic reversibility. Annealing at 1200°C resulted in recrystallization.

The significance of the hcp phase, its relation to the observed $\{111\}_A$ habit, and the effect of stress will be discussed with respect to the mechanism of the transformation.

Study of Isothermal Transformations of Austenite by Thermionic Emission Electron Microscopy: S. R. Rouze and W. L. Grube, General Motors Corp.

The isothermal transformation of austenite to pearlite and to bainite has been investigated utilizing thermionic emission electron microscopy. The growth habits and morphology of the products formed, and the rates of transformation, in plain carbon and alloy steels have been compared by simultaneous cinematic recording of both time and the transformation as it proceeds at temperature in the thermionic emission microscope. In alloy steels, the austenite → bainite transformation has been observed and recorded at temperatures as low as 450°C.

The rate of advance of the transformation fronts depends, as expected, upon the degree of undercooling below the critical temperature and the alloy content. In plain carbon steels the pearlite transformation fronts proceed at rates in the neighborhood of 50 μ per sec. For the bainite reaction, it is observed that bainite colonies broaden by the successive addition of needles. It appears that each needle completes its growth before the next one is added. In plain carbon steels both the growth rate of the primary needles and the rate of broadening of the colony is markedly faster than the corresponding growths in alloy steels, but the ratio of the two rates appears to be independent of alloy content.

While twin bands and grain boundaries have little effect upon the advance of a pearlite transformation front, a bainite front seldom crosses austenite grain boundaries due to the crystallographic orientation dependence of the bainite transformation mechanism. When a bainite needle, in the course of growth, bridges a grain boundary or intercepts a coherent twin boundary, marked *refraction* occurs.

A motion picture film showing the austenite → pearlite and the austenite → bainite transformations as they occur in the thermionic emission microscope will be presented.

On the Reduction in Tensile Strength and the Stress-induced Martensitic Transformation of Some Austenitic Stainless Steels at Very Low Temperatures: R. P. Reed, C. J. Guntner, and R. L. Greeson, National Bureau of Standards.

An anomalous decrease in the tensile and notch tensile strength of annealed austenitic steels type 304 (18 Cr-8 Ni), type 202 (17 Cr, 5 Ni, 8 Mn), and type 350 (16 Cr, 4.5 Ni, 3 Mo) has been found at 20° or 4°K. In some alloys the decrease was rather drastic, e.g. type 350 decreased from 333 ksi at 76°K to 210 ksi at 20°K. In all cases the ductility also decreased considerably.

In an attempt to determine the nature of the embrittlement, a study has been made of the stress-induced martensitic transformation in these alloys. X-ray and metallographic evidence indicate that two martensitic products are formed: an acicular bct structure and a banded hcp structure. Photomicrographs, which were taken after deforming at low temperatures to various strains, illustrate the deformation characteristics. The percentages of the two martensitic products, as determined by X-ray analysis, stain etching, and magnetic measurements are plotted versus strain for alloys type 202 and 304. At a given strain, as the temperature decreases, the percentage of the bct product approaches a limit, while the hcp product continues to form more readily.

Using these results, an interpretation is made pertaining to the decrease in tensile strength and elongation of austenitic steels at very low temperatures.

This work was sponsored by the Advanced Research Projects Agency of the Department of Defense.

Transformation Studies of Gray-Tin Single Crystals: R. G. Wolfson, M. E. Fine, and A. W. Ewald, Northwestern University.

The gray-to-white tin transformation was studied in thin wafers of single-crystal gray tin about 0.015-in. thick and ⅛ in. across. The motion of the transformation interface was recorded by motion-photomicrography in the temperature range 24.2° to 46.5°C.

The linear growth rate is not a single-valued function of the temperature. Imposed upon the strong temperature dependence of the average rate is a variation which is well defined at a given temperature; the spread in the observed values decreases sharply with increasing temperature. Near room temperature, the average rate is of the order of 0.1 micron per sec, and the variation corresponds to a factor of about 150; at 46.5°C, the average rate is about 40 microns per sec, and the variation is only a factor of 2.

The variation is attributed to two observed stress-relaxation processes: cleavage in the gray tin and plastic deformation accompanied by recrystallization in the white tin. It seems likely that the gray-to-white tin transformation proceeds by a coordinated movement of the atoms across the interface, and that the maximum linear rate is limited by the release of stored energy through the recrystallization of the white tin.

This research was supported by the Air Force Office of Scientific Research under Contract No. AF49(638)-524.

Observations of the Physical Damage Resulting From Thermal Cycling Plutonium Through Its Low Temperature Phase Transformations: R. D. Nelson, General Electric Co.

Thermal cycling plutonium through its allotropic transformations resulted in extensive physical damage. This was shown primarily by a density decrease as a function of the number of cycles. Thermally-cycled specimens contained voids and macrovoids which were observed metallographically. The voids initially appeared as internal microcracks and upon repeated thermal cycling became somewhat symmetrical. After repeated cycling the distribution of the voids was generally quite uniform except at the specimen ends, where the voids were larger than those in the interior. Also, the voids were randomly oriented throughout the samples. The experimental observations of cycling damage in plutonium were: (1) The physical damage resulting from thermal cycling plutonium through its allotropic transformations was greater than for any other metal. (2) The extent of physical damage varied considerably depending on the cycling conditions and the characteristics of the metal, particularly inclusions and casting imperfections. Specimens containing a larger number of inclusions and casting imperfections showed more extensive void formation. (3) The extent of physical damage, determined by density measurements, was directly proportional to the volume change associated with the allotropic transformations. (4) The presence of voids caused a decrease in mechanical properties. The tensile strength of specimens containing 5 to 7 pct voids decreased from 62,800 to 30,000 psi and the yield strength from 39,300 to 26,400 psi. (5) Increasing the specimen size resulted in a greater decrease in density as a function of the number of cycles. (6) Cycling rate may have affected the extent of void formation but probably not to the degree that specimen size and impurities did. (7) Varying the length-to-diameter ratio of rods having a constant diameter had little or no effect on the extent of void formation. (8) Bending of rods always occurred in thermally cycled rods. (9) Radial and longitudinal growth occurred in all of the cylindrical specimens that were thermally cycled. A depression also appeared in the ends of each rod. (10) Fracture frequently occurred when specimens were cycled. (11) Voids collapsed when thermally-cycled specimens were subsequently allowed to transform under 5000 psi.

Process Simulation

9:00 am to 12:00 m, Pennsylvania Room-East

(Joint Session Sponsored by Iron and Steel Division and Extractive Metallurgy Division)

Chairmen: H. W. Meyer, Jones & Laughlin Steel Corp., and J. R. Dietz, National Steel Corp.

Predicting Effects of Oxygen, Moisture, and Fuel Additions on Blast Furnace Operations With a Computer: A. L. Hodge, Linde Co.

Use of Regression Analysis as Applied to Simultaneous

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Equation Models: D. C. McCune, Jones & Laughlin Steel Corp.

A Simulation of Melt Shop Operations Using a Computer: R. Neate and W. J. Dacey, Steel Co. of Wales, Ltd.

Application of Small Electronic Computers to Pyrometallurgical Research: R. J. Leary, U.S. Bureau of Mines.

Effects of Extremely High Pressures

9:00 am to 12:00 m, Hall of Flags

(Sponsored by IMD Chemistry and Physics of Metals Committee)

Chairman: S. S. Brenner, General Electric Co., and L. Kaufman, Manufacturing Laboratories, Inc.

Static Pressure Research in Metallurgy: J. E. Hilliard, General Electric Co.

Phase Transitions and Melting Temperatures of Pure Metals at High Pressures: G. C. Kennedy, University of California.

Use of Shock Waves to Study High Pressure Effects: G. R. Cowan, E. I. duPont de Nemours and Co.

Titanium Versus Steel and Aluminum in Pressure Vessels for Hydrospace, Aerospace, and Space

2:00 to 5:00 pm, West Ballroom

(Sponsored by IMD Titanium Committee)

Chairman: R. I. Jaffee, Battelle Memorial Institute

Film: "The March of Titanium," produced by Titanium Metals Corp. of America.

Procedures for the Evaluation of Fracture Toughness of Pressure Vessel Materials: W. S. Pellini and J. E. Srawley, U.S. Naval Research Laboratory.

Normal design procedures for internally and externally pressurized pressure vessels are based on criteria of failure by general yielding or instability. Such procedures are valid if the fracture toughness of the material, as fabricated, is sufficient to prevent failure by a fracture mode. The degree of fracture toughness that is required is known to vary with the design, service conditions, and method of fabrication. Because of the complexity of the interactions of these factors the specific fracture toughness requirement is ordinarily determined by testing of models and of full scale vessels with correlation to laboratory fracture tests. The requirements for submarine hull structures have been determined by explosion tests of weldments and are defined as an extremely tough condition associated with the restricted propagation of shear tearing fractures. Charpy V tests have provided effective laboratory specimen correlations for steels of low- and intermediate-strength levels. For materials of higher yield strength to density ratios it appears that the level of toughness associated with full shearing decreases; this may prove to be a limiting factor, as yet the situation is unresolved. Thin high-strength materials for airframes and rockets are evaluated quite differently, most commonly by direct determination of the effect of cracks on fracture stress—a tiny crack in a very high strength material can induce fracture at a fraction of the yield strength. Stress analysis methods are employed to derive values of a characteristic fracture toughness property through which, ideally, fracture stress may be related to flaw size. Temperature transitions do occur but, again, full shear toughness diminishes with increasing strength level. If a very small maximum flaw size can be guaranteed, it is possible to use a material of very high yield strength below its full shear temperature.

Pressure Vessels for Space: V. W. Cooke and R. C. Powell, Pratt and Whitney Aircraft Company.

Design requirements are stated, with yield strength-density ratios considerably exceeding 1,000,000 as an immediate goal. Properties of various materials such as low alloy and stainless steels, aluminum and titanium alloys, and fiber-glass composites are described briefly as used in current pressure vessels of both liquid and solid fueled types. Low alloy steels are described as being strong contenders for highest strength-weight ratios in the future, but it is emphasized that such levels will require a processing technique not yet known as a production method i.e., austforming or variations thereof.

The all-β titanium alloy, B-120VCA, is stated as showing one of the highest strength-weight ratios yet obtained in a full scale, all-metal pressure vessel. Burst test failure of a full scale vessel is described and slides shown. Tensile properties of such a vessel are listed as UTS 185,000 to 195,000 psi, YS 175,000 to 185,000 psi, and elongations of 4-8 pct. Gc fracture toughness values were obtained as high as 900. This alloy is noted to have low ductility in the subzero ranges, and therefore not considered suitable for cryogenic service, but it could be ideal for ambient temperature storage of solid or liquid fuels where external corrosion might be a problem, i.e., marine atmospheres. A strong point in favor of B-120VCA is that it has been produced and fabricated by existing production facilities, and therefore its possible use in the immediate future is quite realistic, as contrasted with austforming of steels. Considerable hope is given that this alloy can be pushed to a usable yield strength of 210,000 psi, based on data obtained from subscale pressure tests.

It is noted that titanium alloys have not been brought into use in composite structures of wire or ribbon-wrapped designs, however, this could become just as feasible as use of fiber glass or steel piano wire. It is concluded that no one alloy is the answer to the designer's request for an ideal pressure vessel material, but that several of the titanium alloys are strong possibilities, considering their early stage of development.

Structural Significance of Ductility in Aerospace Pressure Vessels: George Gerard, New York University.

The relatively great proportion of aerospace frames in the form of tension structures has focussed considerable attention upon the attractive strength/weight potential of limited ductility, high strength materials. The successful use of such materials in tension structures which invariably contain stress concentrations requires an appreciation of how ductility acts to reduce such stress concentrations.

The results reported in this paper include an exploratory experimental study of small gage length fracture strains at stress concentrations and an analysis of the weakening and strengthening effects of stress concentrations in terms of a ductility ratio. The structural significance of tests used to evaluate ductility in the presence of stress concentrations are discussed and a procedure to evaluate the ductility ratio as a basic material property is suggested. Finally, the structural strength/weight characteristics of various high strength sheet materials are evaluated in terms of the structural design problem associated with aerospace pressure vessels.

Pressure Vessels for Hydrospace: George Sorkin, Department of the Navy.

The characteristics of titanium and its alloys have been evaluated under the conditions of loading and environment peculiar to Submarine Service. Considering corrosion, density, modulus and yield strength only, titanium alloys are superior to most other structural materials. Other parameters such as fabricability, weldability, notch toughness, transition temperature, resistance to explosion loading and creep are discussed.

Panel Discussion on Mechanical and Metallurgical Principles Affecting Hot Extrusion

2:00 to 5:00 pm, East Ballroom

(Sponsored by IMD Non-Ferrous Metallurgy Committee)

Introduction: Paul Loewenstein and Arthur Geary, Nuclear Metals, Inc.

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Brief presentations on hot extrusion of
Lead: F. A. C. Shaw, American Smelting and Refining Company.
Aluminum: M. E. Molitor, Kaiser Aluminum & Chemical Corporation.
Magnesium: (Speaker to be announced.)
Copper: T. S. Howald, Chase Brass & Copper Company.
Nuclear Materials: A. White, Nuclear Metals, Inc.
Summary: Paul Lowenstein, Nuclear Metals, Inc.

Discussion Period

Theory and Application of Physical Chemistry

(Sponsored by the Iron and Steel Division)

2:00 to 5:00 pm, Pennsylvania Room-West

Chairmen: C. W. McCoy, Crucible Steel Corp. of America, and F. D. Delve, Jones & Laughlin Steel Corp.

Technology of Dwight-Lloyd McWane Process for Making Iron: T. E. Ban, McDowell Co., Inc., and B. W. Worthington, McWane Cast Iron Pipe Co.

Kinetics of the Iron Oxide Reduction Steps: A. Wilhelem, and G. St. Pierre, Ohio State University.

Sulfide Inclusions in Steel: Lawrence Van Vlack, D. K. Riegger, R. J. Warrick, and J. M. Dahl, University of Michigan.

Phase Equilibrium Studies of Synthetic Nonmetallic Inclusions in the FeO-MnO-MnS-SiO_2 System: E. H. Silverman, U. S. Steel Corp.

Effect of Chromium on the Activity of Sulfur in Liquid Iron: N. R. Griffing, Linde Co., and G. W. Healy, Union Carbide Metals Co.

Effects of Extremely High Pressures

2:00 to 5:00 pm, Hall of Flags

(Based on Submission of Research Abstracts)

Chairmen: S. S. Brenner, General Electric Co., and L. Kaufman, Manufacturing Laboratories, Inc.

Effects of High Pressure on Phase Transformations in the Iron-Carbon System: S. V. Radcliffe, M. Schatz, and S. A. Kulin, Manufacturing Laboratories, Inc.

The effects of a hydrostatic pressure of 50,000 atm on phase equilibria, the isothermal decomposition of austenite, and the tempering of martensite have been studied for a number of commercial plain-carbon steels (0.38-1.1 wt pct C) and high-purity Fe-C alloys (0.08-1.23 wt pct C). At this high pressure, the eutectoid temperature is lowered approximately 60°C below that at 1 atm pressure, in fair agreement with the depression computed from the Clausius-Clapeyron relationship. In addition, the austenite-cementite and austenite-ferrite boundaries are displaced towards lower carbon contents and the eutectoid composition is reduced to approximately 0.25 wt. pct C.

The isothermal transformation of austenite to ferrite-carbide aggregates is retarded by pressure at all temperatures in the range between the eutectoid temperature and m_s. Th retardation is less for pure Fe-C alloys than for plain-carbon steels, in which the diffusion of substitutional elements as well as carbon is involved in the tranformation. The morphology of the products of transformation under high pressure differs considerably from that at atmospheric pressure. For the pure Fe-C alloys, the greatest effect on kinetics and morphology is observed for the lower carbon alloys, which do not contain excess cementite as the result of the displacement of phase equilibria during austenitizing under pressure. The presence of such cementite appears to facilitate the subsequent isothermal transformation of the austenite.

The tempering of martensite over a wide range of tempering temperatures is also retarded under high pressure. In plain carbon steels, the rates of both the first and the third stages of tempering are reduced, whereas for pure Fe-C alloys the principal effect of pressure is on the third stage of tempering i.e. the solution of ϵ carbide and the formation of cementite.

On the Pressure Induced Phase Transition Associated With Intense Shock in Iron: P. C. Johnson, B. A. Stein, and R. S. Davis, Arthur D. Little, Inc.

The two-wave structure that is observed in iron subjected to shock pressures greater than 130 kbars at ambient temperatures has been rationalized in terms of a pressure induced $\alpha \rightarrow \epsilon$ transformation. The influence of temperature on the pressure for the transformation has been studied at 80°K to 700°K with a relatively simple technique that depends on the micro-structural change in the steel associated with the transformation and the multiple-pellet technique for measuring shock pressures.

The details of the technique and the observation will be discussed.

Detection of Phase Changes in Iron by Differential Thermal Conductivity Analysis: W. F. Claussen, General Electric Co.

The detection of the α to γ phase changes in iron have been investigated up to 75,000 atm, by means of a new technique termed differential thermal conductivity analysis (DTCA). This method involves the property of thermal conductivity, while the more familiar differential thermal analysis (DTA) involves latent heat changes. A piece of iron and a piece of reference material (nickel) are placed in identical temperature gradients. With a thermocouple at the midpoint of each piece, the difference temperature (ΔT) between the two pieces is recorded by bucking one thermocouple against the other. ΔT is zero if each piece is homogeneous. However, with slowly rising temperature when one piece (iron) becomes inhomogeneous with respect to thermal conductivity in going from the α to the γ phase, ΔT departs from zero. Upon completion of the phase change with rising temperature, ΔT again returns to zero.

The temperature for the α-γ iron phase transformation was found to drop continuously when the pressure is increased, the value at 75,000 atm being 605°C.

The reverse transformation $\gamma \rightarrow \alpha$ was found to be the 10°-40° lower than the forward one. At this time it is uncertain whether this hysteresis is due to poor pressure equalization within the cell or due to a sluggishness in the transformation.

The DTCA method is especially adaptable for ceramic or certain metallic transformations where electrical conductivity cannot easily be used to follow the phase changes.

Compressibilities and Phase Transitions in Solidified Gases at Low Temperatures: J. W. Stewart, University of Virginia.

We have determined PV relations and phase transitions for a number of solidified gases at pressures up to 20,000 atm between 4° and 200°K. Pressure is applied at low temperature by the direct piston displacement method. The solidified gas samples used are believed to be sufficiently plastic so that approximately hydrostatic pressure is transmitted. Volume changes and discontinuities are observed through motion of the piston. The values of compressibility are obtained by the method developed by Bridgman for solid substances. The low temperature high pressure apparatus and results for solid H_2S, SiF_4, SiH_4, NCl, and other substances will be discussed. Transition pressures for phase transformations are determined as a function of pressure. Friction in the high pressure cylinders prevents accurate determination of the volume discontinuity at transitions.

Recrystallization Under High Pressure: L. E. Tanner and S. A. Kulin, Manufacturing Laboratories Inc.

The effects of a hydrostatic pressure of 50,000 atm on annealing behavior have been studied for a cold-worked copper—30 pct Zn

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alloy of commercial purity and high-purity copper using metallographic, microhardness and X-ray diffraction techniques.

At this high pressure, the recrystallization process in both materials is retarded, resulting in higher recrystallization temperatures. Thus, for the alloy, recrystallization was observed to be incomplete after annealing under high pressure at temperatures up to 500°C for periods up to 10 min, which are sufficient to allow full recrystallization at atmospheric pressure. Although annealing under high pressure at 600°C and above gave complete recrystallization within such a period, the grain size was smaller than observed after similar annealing at atmospheric pressure. Moreover, the hardness did not decrease as much, even after the annealing under pressure was prolonged to give a grain size coarser than that observed after annealing at atmospheric pressure. For high-purity copper, the shapes of the X-ray diffraction peaks after recrystallization under high pressure were significantly more diffuse than those from copper recrystallized at atmospheric pressure.

These results indicate that under high hydrostatic pressure, atomic mobility is markedly decreased and that, possibly, a recrystallized structure containing more imperfections is developed.

Effect of Pressure on the Age-hardening Characteristics of a Copper-Beryllium-Nickel Alloy: V. A. Phillips, General Electric Co.

The effect of pressure during heat-treatment on the optical microstructure and microhardness of a commercial copper—2.1 pct Be—0.4 pct Ni alloy has been studied. It was found that the application of 69 to 75 katm pressure during aging at 390°C decreased the growth rate of the discontinuous precipitate nodules by a factor of about 18 from 1.4 to 0.08 μ per min. Microhardness tests indicated that the rate of general precipitation was retarded by a factor of about five without much effect on the peak hardness. Pronounced localized precipitation occurred along slip bands when aging under high pressure.

Calculation shows that the application of 75 katm at the solution temperature of 825°C should reduce the solubility of beryllium in copper to about a half of its normal value. Qualitative experiments indicated that the solubility is substantially reduced since several volume pct of a second phase were present after solution treatment, and furthermore the age-hardenability was comparatively slight. The application of high pressure during solution treatment also gave an unusually fine grain size attributable partly to the inhibition of grain growth by dispersed particles and partly to a reduced self-diffusion rate due to reduction in the equilibrium vacancy concentration.

This research was supported in whole or in part by the U.S. Air Force under Contract No. AF-33(616)-5995, monitored by the Materials Laboratory, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

X-ray Diffraction by Substances at High Pressures: J. S. Kasper, General Electric Co.

There is an increasing need for structural information for substances at high pressures, particularly in the case of reversible phase transformations. Unfortunately, the usual high pressure apparatus precludes obtaining X-ray diffraction measurements. Accordingly, we have developed a technique, first employed by Lawson and coworkers, utilizing a single crystal diamond cell as the pressure chamber.

Diamonds of 2 to 3 carats, in the form of rhombic dodecahedra, have been used to attain pressures up to 35,000 atm. The specimen is contained in a cylindrical hole (about 0.015 in. diam) which is drilled through the diamond. The axis of the cylindrical hole is made to be in the [100] direction. Pistons of hardened steel or of Carbaloy have been used, and the force is applied by compression of a steel spring. MoK_α has been found to be the only practicable radiation for obtaining diffraction patterns. Auxiliary equipment includes dial gauges for reading piston displacements, arrangements for resistance measurements, and a simple resistance furnace for heating specimens up to 500°C.

Various alkali halides which transform from NaCl to CsCl structures have been studied, mainly for the observation of the transformed structure. Other substances including graphite and MoS_2 have been investigated for possible transformations and for obtaining compressibility data. In general, for materials of not too high absorption, the cylinder is filled only with the material to be investigated. The diffraction patterns, then, are quite satisfactory.

The emphasis of the work, however, has been on bismuth and its alloys. The high absorption here has required special techniques for successful recording of diffraction patterns. The main feature is the use of thin foils (~0.001 in.) imbedded in zinc stearate as a pressure transmitting medium. Successful recordings of the patterns of transformed bismuth at pressures above 25,000 atm have been obtained. The pattern are complex, however, and a solution of structure has not been made at the time of writing. It has been established also that an intermetallic compound BiSn is formed from the immiscible elements by pressures in excess of 15,000 atm. This pattern too is complex and has not been successfully interpreted.

This research was supported in whole or in part by the United States Air Force under Contract No. AF 33(616)-5995, monitored by the Materials Laboratory, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

Residual Effects of Hydrostatic Pressure on X-ray Diffraction of Aluminum Single Crystals: R. E. Zumwalt, L. P. Skolnick, and J. R. Ferron, University of Delaware.

Single crystal samples of aluminum were prepared so that the (111) planes were exposed at the surface. These samples were soaked in methanol at a hydrostatic pressure of 200,000 psi for a period of about a week.

A careful X-ray diffraction study of these crystals was made before and after pressure treatment; this study involved the measurement of lattice constant and integral line breadth, as well as a Fourier analysis of the line profiles. These studies disclosed a decreased lattice constant and an increased dislocation density at the surface of the treated samples, and an even further decreased lattice constant and a decreased dislocation density beneath the surface. Little change was noted in the lattice strains which were deduced from the Fourier analysis.

The results have been explained by the movement of a relatively few dislocations to the surface of the crystal under the influence of a force field due to the hydrostatic pressure, and the pile-up or accumulation of some of these dislocations at the surface. The results for samples treated 7 days and a sample treated 1 day were essentially the same.

Effect of Annealing at High Hydrostatic Pressures on Dislocations in Lithium Fluoride, Aluminum, Copper, and Iron: J. E. Hilliard, J. M. Lommel, J. B. Hudson, D. F. Stein and J. D. Livingston, General Electric Co.

Following the work of Zumwalt (M.Ch.E. Thesis, University of Delaware, 1960), an investigation is being made of the effect of high-hydrostatic pressures on the density and mobility of dislocations. Single crystals of lithium fluoride, aluminum, copper, and iron have been held for periods of up to one hour at 40 kbars and 550°-600°C. The dislocation structure was examined in aluminum by the Berg-Barrett X-ray technique, and in the other materials by etch pitting.

The high-pressure treatments produced no observable change in the dislocation densities of any of the four materials. More detailed observations were possible on the lithium fluoride than on the other materials because the specimens were prepared by cleaving a crystal in two, and a direct comparison could be made between the cleaved face of the pressure-treated specimen and that of the untreated specimen. It could thus be observed that no movement of the dislocations had occurred during the pressure anneal; whereas, appreciable migration of dislocations took place in a crystal given the same thermal treatment at atmospheric pressure. We thus conclude that, for lithium fluoride at least, the application of hydrostatic pressure decreased the mobility of dislocations.

This research was supported in whole or in part by the United States Air Force under Contract No. AF 33 (616)-5995, monitored by the Materials Laboratory, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

Deformation of Aluminum and Copper Single Crystals Under Large Dynamic Pressures: C. M. Glass, Ballistic Research Laboratories.

Explosive loading has been used to study the effect of high dynamic pressures on aluminum and copper single crystals. Hollow single crystal cylinders have been loaded internally and studied as to: a) the velocity of the high pressure pulse through the material as a function of initial orientation of the specimens: b) the time at which non-symetrical deformation begins in the expansion of the cylinders as a function of initial orientation and pressure at the free surface; c) fracture modes as a function of orientation of the specimens.

Single crystals of copper have been loaded with plane, high-pressure deformation wave generated by explosives. The experi-

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mental arrangement used permits the passage of a compressive wave through a solid specimen, with no tensile reflections from free-surfaces. The specimens have then been analysed for a) change in dislocation density through etch pit techniques b) nature of the strain markings produced, and c) twinning.

The results of the experiments are reported, and a discussion is given concerning the metal reactions in view of current theories on the nature of the large compressive waves generated by explosive loading of metals.

Additional Data on the Shock-induced Transition in Iron-base Binary and Ternary Alloys of Chromium and/or Nickel: E. G. Zukas, F. S. Minshall, and C. M. Fowler, Los Alamos Scientific Laboratory.

The discovery of a shock-induced transition in iron was reported by Bancroft, Peterson, and Minshall in 1956. The metallographic effects of the transition on iron and iron alloys have been studied by numerous experimenters. Reports and bibliographies of recent work in that field were given at the conference on the *Response of Metals to High Velocity Deformation* held at Estes Park, Colorado. At that meeting C. M. Fowler reported on the Hugoniots of some binary and ternary alloys of Fe-Cr-Ni. The purpose of this paper is to present recent data on the surface generated when the pressure of the transition is plotted as a function of the three constituents. Because this is a report of work in progress, features of the surface may become clearer as new experiments are made. However, at present, the surface is as follows: The transition pressure of a standard 1-in. thick target plate of Armco iron is about 128 kbars. Along the zero per cent chromium line, as the content of nickel is increased, the pressure of the transition monotonically decreases, being about 102 kbars at 21.5 pct Ni. Along the 0 pct Ni line, as the content of chromium is increased, the pressure of the transition remains essentially constant until the chromium has reached 5 pct decreases to about 126 kbars in the 7.5 pct to 11 pct Cr range, and finally rises steeply to 180 kbars at 21.5 pct Cr. Several transition pressure points have been obtained for mixed Fe-Cr-Ni alloys, and a rough idea of the surface will be presented. One interesting feature is that the pressure of the transition drops more rapidly when both chromium and nickel are added to the iron, dropping to about 30 kbars for an alloy of 16 pct Cr, 8.3 pct Ni, and balance Fe.

This work was performed under the auspices of the U. S. Atomic Energy Commission.

FALL DINNER

Tuesday, October 18

7:00 pm, Open Cocktail Party
Constitution Room

7:30 pm, Annual Fall Dinner
Pennsylvania Room

Toastmaster: J. H. Jackson, Vice-Chairman, Institute of Metals Division.

Welcome: Leopold Pessel, Chairman, Philadelphia Section, AIME.

Speaker: To be announced.

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Diffusion Controlled Processes

9:00 to 10:30 am, West Ballroom

(Based on Submission of Research Abstracts)

Chairmen: H. W. Paxton, Carnegie Institute of Technology, and J. L. Rutherford, Franklin Institute Laboratories.

Technique for Observing Grain Boundary Migration in Lead: J. L. Walter, General Electric Co.

Studies of recrystallization and grain growth by methods that involve repeated heating and cooling may not give a true picture of rates of boundary migration and of interactions between grains. The grains may become deformed as a result of rapid changes of temperature during heating and cooling.

A technique for the direct observation of grain boundary migration in lead at constant temperature has been developed. Using this technique, one can measure rates of grain boundary migration, observe the formation and disappearance of grain boundary grooves, the formation of growth twins, and the growth of new grains from newly deformed regions.

The lead used in this study contained less than about 0.01 pct impurities. Samples, cold rolled to about 0.030 in. thick, were mechanically polished with soft paper. After polishing, the samples were placed in a small glass dish containing a solution consisting of 80 pct acetic acid and 20 pct hydrogen peroxide. Once the oxidized surface layers had been removed, it was possible to see the grain boundaries and to observe grain boundary migration at room temperature by means of an ordinary metallurgical microscope. The solution acted as an annealing atmosphere and protected the sample from further oxidation.

Rates of grain boundary migration as high as 0.02 m. per minute have been observed by heating the solution to a temperature of 45°C.

Motion pictures have been made, showing the spasmodic nature of boundary migration and the growth of new grains from heavily deformed regions of the sample.

Grain Boundary Relaxation in an Aluminum-Aluminum Oxide SAP-type Alloy: G. S. Ansell and P. E. Arnold, Rensselaer Polytechnic Institute

The grain boundary relaxation of an aluminum-aluminum oxide SAP-Type alloy in both the fine-grained as extruded and coarse-grained recrystallized conditions, was investigated by the use of internal friction measurements. A torsion pendulum apparatus with a vibration frequency range from 0.9 to 1.8 cycles per sec was used to measure internal friction in these materials over the temperature range from 70° to 650°F.

An internal friction peak was observed in testing the fine-grained alloy which was not observed for the coarse grained alloy. This peak associated with grain boundary relaxation, was measured at three different frequencies. The activation energy of grain boundary relaxation was determined from this data to be 12,-500 cal per mole. The activation energy received indicates that grain boundary relaxation in this aluminum-aluminum oxide SAP-Type alloys is quite different from grain boundary relaxation in single phase aluminum alloys. A dislocation model is proposed to explain this behavior.

The sponsorship of this research by the National Aeronautics and Space Administration is gratefully acknowledged.

Anelastic Behavior of a Dilute Al-Mg Alloy: B. S. Berry, International Business Machines Corp.

A torsion pendulum with a range of -190°C to 250°C has been built for a study of the internal friction behavior of quenched

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(or cold worked) dilute binary alloys, particularly in the sub-zero temperature range. The object was to detect an internal friction peak of roughly predictable characteristics, arising from the stress induced reorientation of solute atom—vacancy pairs. After quenching a coarse grained Al, 0.5 pct Mg specimen from 400°C, the internal friction exhibited an almost flat-topped maximum ($Q^{-1} = 2 \times 10^{-4}$) extending from −75°C to −45°C (for a frequency of 1 cps.) The curve was essentially reproducible on heating and cooling. Isochronal anneals of 40 min duration showed that a small reduction in the height of the maximum resulted from treatment at 100°C, but temperatures in excess of 200°C were required to remove the maximum completely. Each successive anneal not only reduced the level of the internal friction maximum, but also changed the shape of the curve and increased the temperature range in which the maximum occurred.

It is believed that this behavior results from the presence of a range of defects in the alloy, of which the single magnesium atom-vacancy pair is only a simple example. Further tests are in progress to check the prediction that a simpler behavior will be found in specimens of lower Mg content quenched from lower temperatures.

Effect of "Active" Oxide Particle (NiO) Dispersions on the Oxidation of Copper: J. A. Sartell and C. H. Li, Minneapolis-Honeywell Regulator Co.

Much work has been done on the mechanical properties of disperse systems, but very little is known about either oxidation mechanism or kinetics in these materials. The Cu-NiO system was chosen for this investigation because the oxidation of Cu-Ni alloys has already been studied extensively. The oxidation of hot-pressed, extruded dispersions made from Cu and NiO powders has been studied and a complementary investigation of their mechanical properties has been made.

The multi-layer oxide scale on the Cu-NiO dispersions is nearly identical to that produced on vacuum-cast Cu-Ni alloys of equivalent nickel content. Inert marker observations indicate that this marked similarity also holds for the mechanisms by which the individual oxide layers grow. Only moderate differences have been detected between the oxidation rates for the Cu-NiO and the Cu-Ni alloys. The significance of these observations will be discussed.

Since the oxidation behavior of Cu-NiO dispersions is similar to the Cu-Ni alloys rather than to pure Cu, it is apparent that the NiO particles are not inert, but play an active part in the oxidation process. To assess the importance of this factor, the mechanical properties of the Cu-NiO materials will be compared with those of $Cu-Al_2O_3$ dispersions, in which the particles are expected to be somewhat less active.

Oxidation of Thin Metallic Films at Elevated Temperatures. Electron Diffraction Study: Goro Shimaoka, National Research Institute for Metals (Japan).

New specimen-treating adaptor was devised for the electron diffraction study of structure changes of metal surfaces occurring in the reaction gas atmosphere at elevated temperatures.

Polycrystalline films of iron, nickel, chromium and iron-chromium alloy (ab. 50:50) were prepared by vacuum evaporation. These films were heat treated in the electron diffraction camera under the pressures of $10^{-2} \sim 10^{-1}$ mm Hg of air, and were observed by transmission method in situ.

In case of iron films heated in the pressure of $\sim 10^{-1}$ mm Hg of air, $\gamma - Fe_2O_3$ or Fe_3O_4 were present at 25° to 350°C, but the Fe_3O_4 decomposed into FeO at 400°C, and FeO, transposed Fe_2O_3 and unknown oxide were observed at 550°C. On the nickel films heated in $\sim 10^{-6}$ mm Hg of air, hexagonal nickel coexisted with cubic nickel up to 350°C, but only cubic nickel was observed above 365°C and no oxide was detected up to 600°C. But for specimens heated in 10^{-2} mm Hg of air, NiO was detected at 600°C. On the chromium films heated in $\sim 10^{-4}$ mm Hg of air, fine crystals of oxide (probably cubic $\gamma - Cr_2O_3$ or Cr_3O_4) at 25° to 500°C, but $\alpha - Cr_2O_3$ crystals were detected at 550° to 600°C. On the Fe–Cr (ab. 50:50) alloy films heated in $\sim 10^{-4}$ mm Hg of air, fine crystals of oxide (probably $\gamma - (Fe, Cr)_2O_3$ or $FeCr_2O_4$) were observed in the temperature range of 25° to 650°C, but $\alpha - (Fe, Cr)_2O_3$ crystals were detected at 670°C. These observations are available for the study of the structure of oxide-base metal interface of some heat-resisting metals.

Surface Graphitization of Iron-Carbon Alloys: G. R. Speich, U.S. Steel Corp.

Formation of graphite at the free surface of two high-purity iron-carbon alloys containing 0.88 pct C and 1.16 pct C during heat treatment in a vacuum has been investigated by hot-stage microscopy and electron diffraction. Surface graphite was observed to form at temperatures below the austenite solubility limit for graphite and above 650°C.

The rate of surface graphitization was found to be markedly dependent on the crystallographic orientation of the underlying austenite. Surfaces approximating (111) planes exhibited the most rapid rate of graphitization. The basal plane of the graphite layer was found to lie parallel to these surfaces. It is suggested that the dependence of the rate of surface graphitization on orientation results from the formation of a low-energy interface between the graphite basal plane and the (111) austenite plane.

Influence of Diffusion of Interstitials on the Usefulness of Ceramic-Refractory Metal Composites at Elevated Temperatures: M. L. Hill, John Hopkins University.

A structural sheet material consisting of alternate layers of a ductile refractory metal and a brittle ceramic oxide or carbide is considered from a hypothetical viewpoint for use in applications involving repeated cycles of heating and cooling. It is assumed that the metal layers will provide ductility or toughness to the *as-fabricated* composite and that the useful life will be limited because of interface reactions which will occur at elevated temperature and which will introduce interstitials into the metal and cause it to become brittle on subsequent cooling. The useful life is arbitrarily defined in terms of the time required for diffusion of the interstitials into the metal layers at elevated temperatures.

The useful life of composites having Tantalum or Columbium layers from 0.01 to 0.001 in. thick between layers of oxides or carbides is estimated to be of the order of 1 to 1000 sec at 2000° to 2500°F. This is too short for most practical applications. It is concluded that there probably are no combinations of ceramic-refractory metals with sufficiently low values of the diffusion coefficient to yield a reasonable *diffusion-controlled* life and that successful development of such composite materials will therefore depend on an understanding of the reactions at the ceramic-metal interface and upon special barrier layers designed to retard the reactions or decrease the concentration of interstitials dissolved in the refractory metal.

Symposium on Metallic Moderators and Cladding Materials

9:00 am to 12:00 m, East Ballroom

(Sponsored by IMD Nuclear Metallurgy Committee)

Chairmen: D. E. Thomas, Westinghouse Electric Corp., and C. E. Lacy, General Electric Co.

Fabrication and Evaluation of Zircaloy-2 and Zircaloy-4 Tubing Used to Clad Uranium Dioxide and Aluminum—Plutonium Fuel: R. C. Aungst, K. M. Haws, and P. L. Farnsworth, General Electric Co.

Fuel elements for the Plutonium Recycle Test Reactor are clusters of 19 uranium dioxide or aluminum-1.65 pct Pu alloy fuel rods clad in Zircaloy-2 or Zircaloy-4. High-quality cladding is required to prevent expensive in-reactor fuel element failures. The quality of Zircaloy-2 and Zircaloy-4 tubing has improved as test and inspection techniques, capable of detecting defective tubes, have been developed. An immersion ultrasonic test, capable of detecting defects as shallow as 0.002 in. on the inner tube surface, has been particularly impressive. Details of this and other quality tests are reported. Observations of the behavior of tubing during swaging indicate that a small grain size is desirable if cracking is to be avoided.

Mechanical Behavior of Cold Worked Nuclear Grade Zircaloy-2 Tubing: H. H. Klepfer and C. N. Spalaris, General Electric Co.

Mechanical behavior of a sample from a large order of nuclear grade Zircaloy-2 tubing was studied as part of an over-all materials program. The chemical, metallographic, and crystallographic nature of the tubing is described and related to mechanical behavior. Data indicating the response in the mechanical properties due to annealing, hydrogen additions and neutron irradiation are presented.

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Development of Zircaloy-4: J. N. Chirigos, S. Kass, W. W. Kirk, and G. J. Salvaggio, Westinghouse Electric Corp.

The corrosion behavior in high-temperature water and steam, the physical and mechanical properties, the irradiation behavior, and the fabrication of Zircaloy-4 are reviewed and these properties are compared with those of Zircaloy-2. In general, Zircaloy-2 and Zircaloy-4 are very similar in behavior except that during hot-water corrosion Zircaloy-4 absorbs less hydrogen than does Zircaloy-2

Creep and Corrosion Behavior of Some High Strength Zirconium Alloys: D. L. Douglass, General Electric Co.

The effects of composition, phase morphology, and heat treatment on creep and tensile properties and on corrosion in high-temperature water and steam have been evaluated for three zirconium ternary systems.

Dilute alloys, containing less than 1 atomic pct total solute, exhibited corrosion resistance equal to or better than that of Zircaloy-2 but had inferior mechanical properties. Mechanical properties were significantly improved by adding 3 to 7 atomic pct solute; however, the added solute caused a decrease in corrosion resistance. The Zr-Sn-Cb system showed the best combination of creep and corrosion behavior.

Zr-Sn-Cr and Zr-Cb-Cr alloys generally were much less corrosion-resistant than Zr-Sn-Cb alloys but could be heat-treated to high strength with considerable improvement in creep resistance.

Zirconium Alloys for Cladding High Temperature Fuel Elements: R. K. Wagner, H. E. Kline, and D. I. Sinizer, Atomics International.

A group of zirconium alloys which exhibit good elevated temperature properties were prepared by standard melting and fabricating techniques. Alloy compositions investigated include zirconium alloyed with aluminum, tin and molybdenum as well as Zircaloy-2 modified with ¼ pct Al and with columbium.

To evaluate the effect of the alloy additions, a variety of mechanical tests were performed over a range of temperatures. A selected group of the alloys were exposed to sodium at 1050° and 1200°F. After a 2500 hr exposure period, the alloys exhibited corrosion characteristics comparable to unalloyed zirconium.

High Temperature Cladding Alloys: J. F. Collins and J. A. McGurty, General Electric Co.

For high temperature applications, fuel element cladding materials are required to protect the base metal structure and to contain the fuel so as to minimize fission product release and fuel loss.

Since the early work on molybdenum clad fuel element concepts, many metallic cladding systems have been investigated. The developments in stainless steel and nickel alloy claddings are discussed and the more recent applications of new oxidation resistant ferrous-base alloys and chromium-base alloys as claddings for refractory metals are presented.

Fabrication and Mechanical Properties of Aluminum Powder Metallurgy Products: E. G. Kendall and W. H. Friske, Atomics International.

Mechanical strength studies have shown that the tensile and creep properties of aluminum powder metallurgy (APM) products are superior to those of conventional wrought aluminum alloys at temperatures above 800°F. APM alloy M257 has been extruded into several longitudinally-finned tubular shapes for the cladding of nuclear fuels. Techniques for joining APM alloys, including a diffusion bonding process developed for making end closures in M257-clad fuel tubes, are described.

Effects of Gases in Metals

9:00 to 10:30 am, Pennsylvania Room East

(Based on Submission of Research Abstracts)

Chairmen: W. D. Robertson, Yale University, and A. Lawley, University of Pennsylvania.

Control of Oxygen in Copper During Refining: W. F. Harris and Joseph Easha, Jr., Westinghouse Electric Corp.

The presence of a defined quantity of oxygen in fire refined tough pitch copper is necessary to satisfy specification requirements. It is essential therefore, that an accurate and rapid method for the determination of oxygen be available for use during the refining operation.

For many years basic control of refining operations depended on a visual observation of small chill specimens poured at various intervals during processing. The *set* of these samples was related to the physical properties of the metal. Since the *set* is dependent on many factors other than oxygen content, this method, while giving a general indication of the condition of the copper, does not accurately relate the oxygen content.

For the last three years a modified vacuum fusion procedure which enables the operator to obtain the oxygen content of the copper within 15 min of sampling has been in use at the Westinghouse Copper Mill. The method consists of melting the copper contained in a graphite boat in an evacuated system, and measuring the amount of CO liberated. With this system the oxygen content of the copper can be accurately followed during poling. The oxygen content of the copper is also followed during pouring so further deoxidation can be accomplished if necessary. The apparatus is also used to study the distribution of oxygen in wire bars and the relationship between the oxygen content of the copper at the time of pouring and the oxygen content of the finished wire bar.

The apparatus for the determination of oxygen in copper will be discussed as well as the results of studies conducted on the distribution of oxygen in wire bar and the variations in oxygen content of the copper during poling and pouring.

Effect of Hydrogen on the Mechanical Properties of Molybdenum and Other Transition Metals: W. Liebmann, Radio Corp. of America, and Alan Lawley and Robert Maddin, University of Pennsylvania.

High purity molybdenum wires have been quenched in vacuum and in a hydrogen atmosphere, from near the melting point. Subsequent changes of mechanical properties on ageing show that serious embrittlement occurs after the hydrogen quench, while the vacuum quench has only a small effect.

The embrittling effect has two distinct components; one of these is strain rate dependent, while the other is not. By combining the mechanisms for hydrogen embrittlement as suggested by Blanchard and Troiano, and by Eustice and Carlson, and by defining the origin of the large interaction energy between a dislocation and a hydrogen atom for the case of the second mechanism, a theory is developed which is not only able to explain the present results for molybdenum, but also gives a more general explanation of the hydrogen embrittlement of various metals.

The character of the hydrogen-metal interaction changes from largely ionic on the left side of the periodic system to largely metallic on the right side of the system. When the interaction is mainly metallic in character, an embrittling mechanism based on Cottrell's theory of brittle fracture is effective; for a largely ionic interaction, embrittlement is caused by direct electrical interaction of the moving dislocation and the hydrogen atom.

The diffusion energy of hydrogen in molybdenum is found to be 0.4 ± 0.02 ev. This value is in good agreement with diffusion energies reported for other transition metals. There is no direct evidence for the presence of quenched-in vacancies, however, the mechanical properties measured may not be sufficiently affected by the presence of vacancies in order that the latter may be detected.

This work was sponsored by the Office of Naval Research.

Observations of the Hydrogen Yield Point in Iron: M. L. Hill, John Hopkins University.

A study was made of the yield point effect in 0.200 in. diam tensile specimens of a vacuum melted iron that had been treated in hydrogen gas at 1840 psi and 580°C and quenched to room temperature in water. This treatment resulted in the introduction of 11 ±0.3 ppm of hydrogen which was uniformly distributed throughout the specimens. Several of the hydrogen-charged specimens were first strained to about 6 pct elongation at −78°C and were then aged for from 1 to 4 hr at 25°C. Following this, the specimens were cooled to −150°C and tested for a yield point. This pattern of straining and aging was employed in an effort to simulate the experiments of Rogers in which a yield point at −150°C was observed in electrolytically charged specimens.

Only a slight discontinuity was found in the load-elongation curves of the pressure-charged specimens when they were tested at −150°C. There was no drop in load and only about 0.1 pct elongation at constant load occurred before the onset of the usual work hardening.

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Experiments were also done in which hydrogen was charged into the same iron by electrolysis, using the same procedure and conditions employed by Rogers. A yield point effect consisting of about 1500 psi drop in load and about 0.3 pct yield point elongation was observed in such specimens at −150°C. Such specimens were found to contain from 4 to 7 ppm of hydrogen as compared to the 11 ppm in the pressure charge specimens. This inverse relationship between the hydrogen content and the magnitude of the yield point effect is believed to be due to a non-uniform distribution of hydrogen which probably existed in the electrolytically charged specimens.

An X-ray Investigation of the Effects of Hydrogen in Iron: A. S. Tetelman, C. N. J. Wagner, and W. D. Robertson, Yale University

X-ray line broadening resulting from the cathodic charging of hydrogen into annealed and cold worked pure iron was investigated. Electrolytic charging at room temperature resulted in a broadening of all reflections, and Fourier analysis showed that the broadening was caused both by distortion and the presence of small, anisotropic domains. The Fourier coefficients, half widths, particle sizes, strains, and recovery kinetics were found to be identical for iron that had been charged with hydrogen and for iron strained 5 pct in tension. The apparent activation energy for recovery of line broadening, following either cathodic charging or a tensile strain of 5 pct is 42.0 ± 5 kcal per mole in the temperature range 400-500°C. No significant change in lattice parameter was observed as a consequence of cathodic charging. All the evidence indicates that the effects of hydrogen on the X-ray diffraction characteristics of pure iron can best be interpreted in terms of plastic deformation, and there is no X-ray evidence to substantiate the recent proposal that hydrogen occupies tetrahedral interstitial positions in the iron lattice.

Trapping Hydrogen in Cold-Worked Steel: H. H. Podgurski, U. S. Steel Corp.

Large amounts of hydrogen are absorbed by cold worked mild steel on exposure to hydrogen gas at 1 atm pressure and 300°C. This hydrogen is not evolved at an appreciable rate in the 25° to 200°C temperature range and only very slowly at 275°C; deuterium behaves similarly. It is found that if steel so charged at 300°C is subsequently pickled at 25°C, the acid-charged hydrogen escapes first (below 275°C!) just as though the hydrogen or deuterium charged at 300°C were absent. The hydrogen absorbed in the 300-400°C range is shown to be present principally as methane; this was extracted quantitatively by a newly developed method involving bromine-methanol dissolution of the steel. Furthermore, the amount of methane is in accord with the equilibrium $2H_2 + FeC = CH_4 + 3Fe$, and is present in microvoids. The microvoid volume, calculated from the methane content via the equation of state for methane, corresponds approximately to that calculated from the measured density decrement produced by cold work. This suggests that only a small fraction of the measured density change or volume increment may be associated with dislocations generated by cold working steel.

Precipitation of Helium Along Dislocations in Aluminum: G. T. Murray, Materials Research Corp.

Helium was generated in an Al, 0.05 atomic pct Li alloy by neutron transmutation of the Li^6 atoms (7.5 pct of natural Li). Metallographic examination after subsequent heating of the irradiated specimens revealed that numerous small cavities were present, presumably due to the precipitation of helium in the form of bubbles. Utilizing a dislocation etch pit reagent it was found that the highest cavity density always occurred in the region of highest dislocation density. Furthermore, a series of cavities appeared to delineate the track of a moving dislocation. In regions of low dislocation density a cavity was often associated with a dislocation etch pit. It is believed that the helium precipitates along the dislocations in the form of continuous or semi-continuous cylindrical cavities and that perhaps the first stage of observed bubble growth is related to the collection of this gas at dislocation network nodes. Subsequent drainage of the atoms along the tunnels to grain boundaries could account for the grain boundary crack formation observed by other investigators.

This work was sponsored by the Atomic Energy Commission, Division of Reactor Development.

Metallographic Study of the Behavior of Helium in Uranium and Uranium Alloys: Alvin Boltax, Westinghouse Eectric Corp.

The behavior of helium in uranium and uranium alloys (U-1.6, 2.8, and 10 weight pct Mo, U-2 weight pct Zr, U-1 and 2 weight pct C, U-3.8 weight pct Si) after various heat treatments was studied by metallographic techniques. Helium concentrations equivalent to the fission gas concentration after 1 atomic pct burnup (gas concentration of about 5 cu cm at stp per cu cm of metal) were obtained by irradiating samples with 31.5 mev α particles (in the MIT cyclotron) to produce a narrow band of helium about 4 mils below the surface of the samples. Following irradiation, the samples were given various isothermal and thermal cycling heat treatments to permit the helium to form gas bubbles. The helium gas bubbles were observed in the light microscope after mechanical polishing and after chemical etching and in the electron microscope after cathodic vacuum etching.

The observations of the helium gas bubbles formed by various heat treatments between 475° to 1075°C will be summarized. The major fraction of the gas bubbles in any sample, independent of heat treatment, were between 0.05 to 2 μ in diam (10^{10} to 10^{14} per cu cm) and can account for up to about 15 pct porosity. Post-irradiation heat treatment above 900°C gives rise to the formation of a small number (about 10^8 per cu cm) of large bubbles between 10 and 150 μ in diam which can lead to swelling of several hundred pct. The large volume increase produced by the latter bubbles is similar to that observed in metallic reactor fuel elements irradiated at temperatures above 400°C. Comparisons of the behavior of cyclotron-irradiated and neutron-irradiated samples will be presented.

Some effects of recrystallization and phase transformations on the behavior of the helium gas bubbles will be described. In addition, a theoretical model for bubble coalescence and tentative explanations for the formation of the large bubbles will be briefly presented.

Work carried out at Nuclear Metals, Inc. under sponsorship of the Atomic Energy Commission.

The Decomposition of Austenite by Diffusional Processes

9:00 am to 12:00 m, Pennsyvania Room-West

(Sponsored by IMD Ferrous Metallurgy Committee)

Chairmen: R. F. Mehl, U. S. Steel Corp.

Theory of Nucleation in Solid-Solid Transformations: J. N. Hobstetter, University of Pennsylvania

Theory of Diffusional Growth in Solid-Solid Transformations: J. S. Kirkaldy, McMaster University

Theory of the Pearlite Reaction: J. W. Cahn and W. C. Hagel, General Electric Co.

The Formation of Pearlite: M. Hillert, Swedish Institute for Metals Research

An Electronmicroscopic Study of the Mechanism of the Pearlite Reaction: L. S. Darken and R. M. Fisher, U. S. Steel Corp.

Physical, Electrical, and Magnetic Properties of Metals and Alloys

9:00 to 10:30 am, Hall of Flags

(Based on Submission of Research Abstracts)

Chairmen: E. Korostoff, University of Pennsylvania, and L. Pessel, Radio Corp. of America

High Conductivity Copper-rich Cu-Zr Alloys: M. J. Saarivirta, Amco Research & Development, Inc.

This investigation determined the solid solubility and effect of zirconium in copper. Some room and high temperature properties of Cu-Zr alloys were also determined and compared with those of Cu-0.4-0.7 pct Cr alloys.

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The solid solubility of zirconium in copper was found to be about 0.15 pct at the eutectic temperature of 980°C. Zirconium increases the resistance of copper to softening, having its greatest effect at about 0.13-0.15 pct Zr. These alloys precipitation harden to a small extent, therefore, cold working contributes more to the strength than does precipitation hardening. The optimum properties are obtained by solution annealing followed by cold working and aging.

Alloys containing 0.10-0.15 pct Zr have 60-70,000 psi tensile strength and 90 pct IACS electrical conductivity at room temperature after solution annealing followed by cold working and aging. At elevated temperatures of 20-600°C cold worked and aged Cu-0.10-0.15 pct Zr alloy has better strength, ductility, and conductivity than does cold worked and aged Cu-0.4-0.7 pct Cr alloys.

Electrical Conductivity of Non-stoichiometric α-Nb_2O_5: E. H. Greener, D. H. Whitmore, and M. E. Fine, Northwestern University

The electrical conductivity of $\alpha - Nb_2O_5$ monocrystalline and sintered specimens, measured under a constant ambient oxygen pressure and over the temperature range 300° to 900°C, exhibits an exponential temperature dependence with an activation energy of 1.65 ev. The isothermal conductivity, σ, of near-stoichiometric $\alpha - Nb_2O_5$ is related to the ambient ovygen partial pressure, P_{O_2} by the power law expression $\sigma = \text{Const.}\ P_{O_2}^{-0.24 \pm 0.01}$.

The electrical conductivity of heavily reduced specimens was also measured in the temperature range 600° to −77°C. For dilute concentrations of defects, the temperature dependence of the conductivity yielded activation energies of approximately 0.5 ev in the temperature range 600° to 100°C and 0.1 ev in the temperature range 100° to −77°C. These energies decreased as the degree of reduction (and consequently the number of defects) increased. For the highest degrees of reduction obtained, the material behaved as a degenerate semiconductor.

These observations may be rationalized on the basis that reduced $\alpha - Nb_2O_5$ becomes a metal-excess, n-type semiconducting oxide containing oxygen vacancies capable of trapping electrons.

A detailed analysis of the relation between conductivity, ambient oxygen pressure and concentrations of oxygen ion vacancies has indicated that the conductivity dependency may be ascribed to a mechanism whereby defect levels (due to oxygen ion vacancies with either one or two trapped electrons) are created and electrons are thermally excited from these levels into the conduction band.

This research was supported by the Department of Navy, Office of Naval Research.

Electrical Resistivity of Liquid Sodium, Liquid Lithium and Dilute Liquid Sodium Solutions: J. F. Freedman, International Business Machines, and W. D. Robertson, Yale University

The three factors of atomic size, ion core potential and charge which contribute to the increment in electrical resistivity produced by solutes in metallic solutions, have been separated and experimentally evaluated in liquid sodium from 100°C to 550°C employing a stainless steel conductivity cell.

The effect of atomic size was investigated in the dilute alkali metal solutions, since these solutions exhibit a maximum difference in atomic radius of 50 pct. This range of atomic size difference is large enough for a direct evaluation of the contribution of dilation to the resistivity increment.

The effect of solute core potential was investigated independently in dilute solutions of silver and gold in liquid sodium. There is sufficient difference between the ion core structures of the two groups to produce an important contribution to the resistivity, in addition to that associated with dilation.

Finally the effect of solute charge (i.e., a screened coulomb potential) was evaluated by measuring the change in resistance of liquid sodium produced by the addition of the multivalent solutes, cadmium, tin and lead.

The results may be expressed in a semi-empirical relationship defining the increment in resistivity of a monovalent solution:

$$\Delta\rho = 0.063\ (\Delta \text{ Atomic Number}) + 0.53\ (\Delta V/V)^2$$

microhm-cm per atomic pct solutes.

where $\Delta V/V$ is the fractional difference in atomic volume of solute and solvent and the first term involves the difference in atomic number of sodium and the solutes lithium, potassium, cesium, rubidium, silver and gold.

The fact that a separation is possible and to some extent justified in terms of ideas and relationships developed for application to solid solutions can be interpreted to mean the most significant feature of the scattering process is the difference between the solute and the surrounding solution, rather than the absence of long range order.

Compressibilities of Liquid Alloys: R. B. Gordon, Yale University, and Gerald Abowitz, Cornell University

The compressibility of a fluid reflects its structure and the strength of the bonding forces between its atoms or molecules. The compressibilities of a number of metallic liquids have been determined from measurements of the velocity of sound in the liquid and its known or measured density. In liquid alloys of Hg with Pb, Sn, Bi, Tl, In, Zn, and Cd, the addition of small amounts of solute metal causes a very rapid decrease in compressibility. This is in contrast with the Na−K and Pb−Sn systems where the compressibility changes only gradually with composition. These same mercury alloys are known to display a decrease in electrical resistivity and to become more paramagnetic as the solute content is increased. It is suggested that liquid mercury retains remnants of the covalent bond network of the mercury crystal and that the addition of small amounts of the above solutes destroys this covalent bonding, allowing the dilute amalgams to assume the structure of a typical metallic liquid.

Lattice Strains and X-ray Stress Measurement: M. J. Donachie, Jr., General Dynamics Corp., and J. T. Norton, Massachusetts Institute of Technology

The anomalous behavior of polycrystalline metals after plastic deformation in tension has, for 20 years, attracted the attention of investigators interested in X-ray stress measurement. In view of the contradictory results reported in the literature, the present investigation was undertaken to further explore the relationship of residual lattice strains produced by plastic extension to residual stresses. In order to study this behavior, it was first necessary to confirm the simple mechanical stress-lattice strain relationships in the elastic range of applied stresses.

Ingot iron and 2024 aluminum sheet were extended uniaxially at stresses less than and greater than the yield strength. Lattice strains were measured at various angles in planes which were perpendicular to the specimen surface and parallel to the longitudinal and transverse surface directions. Two sets of lattice planes were studied for each metal. Subsequent work was performed to mechanically evaluate the residual stress and to determine the variation of residual lattice strain with depth.

The results of the elastic region studies indicated a linearity of lattice strain with sine squared of the angle, ψ, at which the strain was measured. Linearity of applied stress vs. lattice strain was also observed for all sets of lattice planes studied. The X-ray values of the elastic constants (Young's modulus and Poisson ratio) were compared with the macroscopic values. From these results, it was concluded that lattice strains in a polycrystalline metal subjected to stresses within the elastic range behave in a predictable manner corresponding to the strain ellipsoid. The results of studies after plastic extension were not as clear cut. An apparent linearity of lattice strain with $\sin^2 \psi$ was found and this indicated that a residual stress existed. Mechanical measurements did not confirm this indication.

These results are discussed and the significance of the results to X-ray stress measurement is considered.

Magnetization of the Alpha and Sigma Phases in Several Fe-V Alloys: M. V. Nevitt, Argonne National Laboratory

The saturation magnetization, $\sigma_{s.s.}$, and the spontaneous magnetization, $\sigma_{s.s.}$, of several Fe-V alloys have been measured as part of a study of the electronic structures that characterize various phases in transition metal systems. The investigation began with the alloys in the form of the metastable bcc α phase, obtained by quenchng from 1225°C or above. Then the magnetizations were redetermined after a series of anneals at 625°C which yielded the stable sigma phase as the final product. The measurements were made in the temperature range 5° to 297°K by a modification of a method previously described.

For the alloys quenched from high temperature and containing 60 and 53 atomic pct Fe values of $\sigma_{s.s.}$ of 88 cgs units pet g and 60 cgs units per g respectively were determined. These data, together with the data of Fallot, indicate that in the disordered bcc phase the linear relation between saturation moment and concentration of Fe atoms is retained at least to the vicinity of the equiatomic composition.

Annealing for several hours at 625°C lowered the saturation and spontaneous magnetization by about 20 pct. Most of the decrease occurred prior to the observation of sigma phase nucleation by x-ray diffraction or metallographic examination, but the decrease was concurrent with the appearance of $(100)_\alpha$, $(111)_\alpha$, and $(210)_\alpha$

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superlattice lines in the body-centered cubic patterns. This CsCl-type ordering has been previously observed by Philip and Beck.

The σ phase formed by further annealing for several hundred hours at 625°C was found to be ferromagnetic but with magnetizations lower than the disordered α by about a factor of three.

This abstract is based on work performed at the Argonne National Laboratory under the auspices of the U. S. Atomic Energy Commission.

Refractory Metal and Alloy Sheet Program

2:00 to 5:00 pm, West Ballroom

(Sponsored by IMD Refractory Metals Committee)

Chairman: G. M. Ault, NASA

This session will consist of a series of presentations describing the Department of Defense Refractory Metal and Alloy Sheet Program and related industrial and government research and development activities. The objectives and organization of the program will be discussed and brief progress reports will be presented covering the areas of alloy development and selection, coatings, joining, methods of analysis, standardized test procedures, quality specifications, fabrication, etc.

Symposium on Metallic Moderators and Cladding Materials

2:00 to 5:00 pm, East Ballroom

(Sponsored by IMD Nuclear Metallurgy Committee)

Chairmen: M. J. Whitman, U .S. Atomic Energy Commission, and R. B. Gordon, Atomics International

Thermal Diffusion of Hydrogen in Alpha—Delta Zircaloy—2: J. Markowitz, Westinghouse Electric Corp.

A Review of Metal Hydrides for Nuclear Reactor Applications: W. M. Mueller and J. P. Blackledge, Denver Research Institute

Hydrogenous materials are of considerable interest in reactor applications because of the capability of hydrogen to slow down neutrons. Metal hydrides combine high hydrogen retention at elevated temperatures with mechanical properties far exceeding other hydrogenous materials. The requirements for moderators, reflectors, shields and controls are given and the selection of metal hydrides for these applications are reviewed.

Physical Properties of Yttrium Hydride: E. S. Funston, General Electric Co.

Recent developments in yttrium hydride research have made available a material with exceptional high temperature properties. The usefulness of this material has been enhanced by the determination of physical properties necessary for engineering applications.

Physical properties, such as melting point, density, electrical resistivity, specific heat and thermal conductivity, from room temperature to 2000°F are presented. The Y-H equilibrium system is also discussed.

The Zr-H System: J. B. Vetrano and D. F. Atkins, Atomics International

The phase diagram of the Zr-H system in the region of ZrH to ZrH_2 is presented. The diagram is deduced primarily from dissociation pressure-composition-temperature measurements, with supporting X-ray data.

Mechanical Properties of Zirconium Hydride: R. L. Beck and W. M. Mueller, Denver Research Institute.

The mechanical properties of zirconium hydride are presented as functions of temperature and composition. These data are interpreted in terms of the phase relations which exist in the Zr-H system. The discussion describes why only certain compositional regions are of interest for use as nuclear moderators.

Compatibility of Zirconium Hydride and Carbon Dioxide: H. E. Shoemaker, General Atomic

An investigation was conducted to study the stability of unclad zirconium hydride as a reactor moderator in carbon dioxide coolant. The Δ-phase hydride ($ZrH_{1.6}$) had excellent dimensional stability, but suffered as great as 20 pct Hz loss at 1200°F along with severe corrosion. The α-plus-Δ-phase hydride ($ZrH_{0.9-1.0}$) swelled and warped in a temperature gradient, but did not lose much hydrogen and did not corrode severely even at 1200°F.

Cladding of Delta-Phase Zirconium Hydride: E. S. Hodge and S. J. Paprocki, Battelle Memorial Institute

A study was made of the cladding of solid and powdered Δ-phase zirconium hydride in both rod and flat shapes with stainless steel. The program included investigations of metallurgical bonding, both with and without the use of metallic barrier materials. Types 304 and 347 stainless steel were used for cladding material. The intermediate barrier layer materials used were niobium, molybdenum, a combination of copper and molybdenum, and a combination of copper and niobium.

The pressure-bonding techniques, involving the use of gas pressure at elevated temperatures, were employed in this study. Variable times and temperatures with a constant pressure of 10,000 psi were utilized to produce bonding.

In this study, the best results were achieved in cladding Δ-phase zirconium hydride directly with Types 304 or 347 stainless steel. Good bonds were obtained by pressure bonding at 1600°F for 3 or 4 hr subsequent to pressure bonding at 1900°F for 1 to 2 hr at a pressure of 10,000 psi. Partial bonding was achieved between niobium and zirconium hydride and molybdenum and zirconium hydride.

Structural Materials in Cryogenic Design

2:00 to 5:00 pm, Pennsylvania Room-East

(Sponsored by IMD Structural Materials Committee)

Chairmen: J. H. Rizley, Convair, and R. W. Fenn, Jr., The Dow Metal Products Co.

Detail Design Criteria for Cryogenic Tanks—Room Temperature vs. Cryogenic Temperature Design Requirements: R. E. Jacobsen, Boeing Airplane Co.

Detail design criteria for cryogenic tanks will be discussed. Tank design for cryogenic and room temperature application will be compared and similarity and difference will be emphasized. Difficulties in fabrication techniques and full scale testing that are compounded due to the cryogenic application will also be briefly discussed. The detail design criteria to be covered will consist of internal piping requirements slosh and antivortex requirements and fitting and joint details.

Design Criteria for Airborne Cryogenic Tanks: R. Markovich, The Martin Co.

The emphasis is placed on the selection of materials and the insulating requirements of liquid hydrogen tanks. The discussion will also include the basic design requirements of missile tanks and its function as a part of the missile structure.

Structural Materials in Cryogenic Environment: E. W. Johnson, Aluminum Co. of America

Designing equipment for service in the cryogenic range of temperatures is not fundamentally different from designing for service in other ranges. It does, however, require a shifting of emphasis among various design criteria. Mechanical properties of materials change markedly, and in some cases rather abruptly, as the temperature of the materials is lowered into and through the cryogenic range. The high conductivity, low emissivity, low-temperature ductility and moderate cost of aluminum alloys mitigate the designer's problems.

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Behavior of Aerospace Vehicle Materials at Cryogenic Temperatures Ranging Down to −423°F: J. Watson, Convair Astronautics

The properties of a variety of high strength structural alloys are described in terms of their application in aerospace vehicle systems for a series of sub-zero temperatures ranging down to −423°F. Properties of various high-strength metals and alloys are outlined in terms of their metallurgical structure. The effect of cryogenic temperature on these properties is described for specific examples of alloys finding current application in aerospace vehicles. These alloys include both the cold rolled and heat treatable stainless steels; aluminum, nickel and cobalt base alloys; and selected titanium alloys. The various methods of producing high strengths in these alloys is reviewed briefly, and then the effects of these treatments on low temperature properties are described in some detail.

Since aerospace vehicles using cryogenic propellants depend on welded joints for pressure integrity, the effect of welding on metallurgical structure and hence on properties are reviewed, and the low temperature properties of welded joints are discussed.

Effect of Temperature in Fracture Testing of High Strength Sheet Materials: J. E. Srawley, U.S. Naval Research Laboratory

Ideally, testing should provide information which can be used directly in the design of structures. Rational design against fracture takes the form of comparing a characteristic parameter of the stress field in the vicinity of a flaw or crack with an experimentally determined fracture toughness value for the material, which is also dependent on thickness and temperature. Tensile tests of shaply-notched or pre-cracked specimens provide the primary data. Fracture strength and toughness decrease with temperature, in some cases very sharply over a narrow *transition* range with an associated change in fracture mode. This is related to thickness as well as hardness of the material. Illustrative test results for high strength steels, titanium and aluminum alloys are presented.

Screening Tests Suitable for Evaluating the Relative Notch Sensitivity of High Strength Sheet Alloys: W. F. Brown, Jr., National Aeronautics and Space Administration

The production of light weight liquid propellant tanks for rockets of advanced design requires the use of very high strength to density ratio sheet alloys. Present day LOX tanks employ aluminum alloys or stainless steels with yield strength to density ratios at −300°F not above about 900,000. However, materials such as the titanium alloys, are available which have strength to density ratios nearly double this figure. The use of these new alloys would result in a great weight savings and consequent increased payload capability. In addition, in the near future, it is expected that liquid hydrogen will be used as a propellant.

The use of very high strength to weight ratio sheet at −423°F raises serious questions concerning resistance to brittle fracture in the presence of small cracks or other defects. In order to rapidly evaluate the relative merits of different materials and to establish optimum heat treatments, it is desirable to employ a suitable laboratory screening test. The sharp edge notch tensile test appears to be satisfactory in this respect. Data derived in this test in combination with the smooth tensile and yield strength furnish a relative index of merit for various alloy conditions at −423°F. Examples are shown which permit comparison between alloys presently used for LOX tankage and those which are being considered for advanced liquid propellant rockets.

The Decomposition of Austenite by Diffusional Processes

2:00 to 5:00 pm, Pennsylvania Room-West

(Sponsored by IMD Ferrous Metallurgy Committee)

Chairmen: Morris Cohen, Massachusetts Institute of Technology

Elementary Reactions in the Pearlite and Bainite Transformations: R. I. Entin, Central Scientific Research Institute of Ferrous Metallurgy of Moscow

Thermodynamics of the Bainite Reaction: L. Kaufman, Manufacturing Laboratories, Inc., and S. V. Radcliffe and M. Cohen, Massachusetts Institute of Technology

Mechanisms of the Proeutectoid Ferrite and Cementite and the Bainite Reactions: H. I. Aaronson, Ford Motor Co.

Growth Kinetics and Mechanism of the Bainite Reaction: G. R. Speich, U. S. Steel Corp.

Origin of the Surface Relief Effects Accompanying the Formation of Ferrite and Bainite Plates: J. W. Christian, Oxford University

Proeutectoid, Eutectoid, and Peritectoid Transformations in Non-ferrous Alloy Systems: C. W. Spencer, Cornell University, and D. J. Mack, University of Wisconsin.

Plastic Deformation I

2:00 to 5:00 pm, Hall of Flags

(Based on Submission of Research Abstracts)

Chairmen: A. S. Nowick, International Business Machines Corp. and R. J. Stokes, Minneapolis-Honeywell Regulator Co.

Frictional Stress Acting on a Moving Dislocation in an Otherwise Perfect Crystal: Doris Kuhlmann-Wilsdorf, University of Pennsylvania

The problem of the frictional stress suffered by moving dislocations in otherwise perfect crystals is investigated. This is done without calculating the core energies of dislocations, but by considering stresses and strains on the slip plane. The level of frictional stresses obtained is much higher than reported previously. Since common glide dislocations in metals with close packed structures apparently do not suffer significant frictional stresses, mechanisms are discussed which tend to reduce their effect. A new such mechanism is discovered. It is based on the idea that the positions of dislocation axes are not defined with precision, but only within one to a few times the average displacement of the oscillating atoms. The expected result of this, is a depression of the frictional stress for close packed metals even at very low temperatures, almost no effect on dislocations in crystals with diamond structures, and a temperature dependence proportional to $e^{-\text{const } T/T_m}$ for NaCl type salts and, probably, for bcc metals.

Easy Glide and Grain Boundary Effects in Polycrystalline Aluminum: R. L. Fleischer, General Electric Co. and W. F. Hosford, Jr., Massachusetts Institute of Technology

Tensile tests at 4.2° and 295°K have been made on a group of coarse-grained polycrystals in an attempt to bridge the gap in measurements between fine-grained polycrystals on one hand and single crystals and bicrystals on the other.

From X-ray optical, and work hardening observations the number of slip systems was deduced for a sample having four crystals per typical cross section. It was shown that not more than two or three slip systems provided appreciable amounts of slip in an average crystal in this sample. For this specimen and others having up to seven crystals per cross section, easy glide could be observed at 4.2°K but not at 295°K.

It was found that the stress-strain curve was strongly affected by the number of crystals per cross section. By considering that a polycrystal is composed of constrained, interior grains with one flow stress, and less confined, surface grains with a lower flow stress, the observed smooth, continuous increase of strength with n is explained. Flow stress did not increase linearly with the inverse square root of grain diameter at either temperature. It is concluded, therefore, that at low temperatures (1) pile-ups at grain boundaries are not important in determining the flow stress in aluminum and (2) the *grain-size* effect is probably due to surface crystals being weaker because of their reduced constraints and hence is a *relative* size effect.

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Rupture of Surface Films on Aluminum Crystals by Plastic Deformation: R. J. Block and Marvin Metzger, University of Illinois

When a filmed metal is deformed, the dislocations must break the film to pass out of the crystal. It is known that this does not always occur but the factors controlling the extent to which the dislocations break through or pile up have not been clearly demonstrated. In the present work, studies have been made using high-purity aluminum single crystals, anodized and tested in tension in a boric acid solution. The course of film rupture was followed by making simultaneous stress-strain and potential-strain measurements. Potential changes were correlated with independent experiments in which the film was removed from known areas.

The initiation of plastic deformation was accompanied by a distinct rise in the potential. During continuous straining in single slip, a saturation value of potential change was reached. The potential behavior depended upon strain rate in a manner consistent with the measured rate of film repair. Although there was clear qualitative evidence of substantial rupture of all the films tested, it was not possible to determine quantitatively the extents of film rupture and dislocation pileup without special additional calibration procedures.

Experiments in which thicker films were applied or removed during the test indicated that the films produced a definite hardening effect after a certain amount of deformation in single slip although the hardening was not detected immediately after their application. This is in general accord with previous work on the effect of films on the plastic flow of crystals, and is consistent with the presence of substantial dislocation pileup.

This work was supported by the Office of Naval Research.

Latent Hardening in Aluminum: U. F. Kocks, Harvard University

A technique of applying two independent strain components to single crytaals is used to operate alternately various pairs of glide systems on intersecting planes, including those of the conjugate, critical, and cross-slip relationships. Torsion and compression are used on thin-walled prismatic (square or hexagonal) single crystals. Stress-strain curves are measured for both components.

Interactions Between Families of Dislocations in the FCC Lattice: B. H. Kear, Franklin Institute Laboratories

Interactions between families of dislocations gliding simultaneously in different slip systems in the fcc lattice are examined. It is shown that under the appropriate conditions various types of small-angle boundary, representing low-energy configurations for the dislocations, are likely to form directly during plastic deformation. The low-energy boundaries may be pure tilt, pure twist, or a combination of pure tilt and pure twist, depending on the orientation in which interactions between the dislocations occur and on their Burgers vectors. The energies of the different boundaries are calculated and the changes accompanying annealing are discussed. It is expected that these boundaries will be formed during the early stages of plastic deformation under the appropriate conditions, but that eventually with increasing stress the boundaries will be broken up, and the dislocations will cut through one another. In this case, point defects will be generated, and analysis shows that the jogged dislocations from one type of boundary become sources of vacancies, whereas in another type they become sources of interstitial atoms.

Microcreep and the Elastic After-effect in Zinc Single Crystals: J. M. Roberts, William March Rice University, and Norman Brown, University of Pennsylvania

Microcreep was observed in zinc single crystals at 295° and 223° A deformed in simple shear. At stress levels below the macroscopic yield point, the creep rate approached zero in a short time, and the initial creep rate varied linearly with stress. After a prior strain, the stress was relaxed below the flow stress, and strain recovery was observed at various stress levels. The rate of strain recovery, in general, varied logarithmically. During the time of stress relaxation after a prior strain, there was also an instantaneous, non-elastic strain recovery.

The strain recovery behavior was very dependent upon the state of the surface of the specimen. The pile-up of dislocations at the surface of the specimen greatly influences the amount of strain recovery that is observed. If a specimen were etched during the period of strain recovery, then the single crystal would strain recover less. A copper coating enhanced the amount of strain recovery as well as effecting the very initial stage of the stress-strain curve. These results, in general, are the same as earlier observations of Barrett, who used single and polycrystalline iron and zinc in torsion. It is also believed that part of the strain recovery is produced by the elastic repulsion of dislocations in the interior of the specimen.

The AEC supported this work.

Easy Glide of Lead Single Crystals: R. L. Fleischer, General Electric Co.

Effects of temperature and impurity content on the easy glide of lead have been examined. Melt-grown crystals of 99.9998 pct lead or lead plus small amounts of tin, copper, or aluminum were deformed in tension at 77° or 4.2°K.

It was found that pure lead shows the three stages and the orientation dependence normally found for fcc crystals, although, as in aluminum, stage II is not linear. Soluble impurity additions extend easy glide; insoluble ones tend to terminate it.

The above impurity effects are interpreted using a hardening model due to Diehl. It is suggested that soluble impurities encourage easy glide by raising the yield stress, and hence the stress to cause secondary slip. The work hardening slope in easy glide is thereby reduced, and the stress at the end of easy glide is increased. It could not be determined whether an additional effect, due to latent hardening, was also present, one complication being the inevitable presence of grip effects, although these might be separated from the others by varying the sample size.

The effects of insoluble impurities are interpreted as due to local stresses increasing secondary slip and therefore work hardening, thus suppressing easy glide. It is suggested that the usual orientation dependence and lack of easy glide previously observed by Feltham and Meakin were due to such impurities in the lead used.

An anomaly was the finding that creep could not be observed at 4.2°K, indicating that, if present, creep is at least an order of magnitude less rapid for lead than for aluminum at 4.2°K.

Plastic Deformation of Lead Single Crystals from 4.2° to 273°K: G. F. Bolling, L. E. Hays, and H. W. Wiedersich, Westinghouse Electric Corp.

Single crystals of zone-refined lead were grown from the melt. These were deformed in tension at temperatures in the range 4.2° to 273°K. The effect of strain rate was examined over four orders of magnitude at 77°K. In common with other fcc metals the analysis of this tensile data displayed at least three distinct stages in the deformation of lead single crystals. Special attention was paid to the onset of the third stage at the stress τ_{III}, and the temperature dependence of this stress was used to estimate the stacking-fault energy.

For certain crystal orientations two distinct linear regions other than easy glide were evident. An examination of other fcc metals also revealed these two stages. The second linear region may be considered a part of the third stage of deformation and an explanation of the phenomenon is offered on this basis.

X-ray Study of Deformation Stacking Faults at Low Temperatures in Lead, Some Lead Alloys, and Aluminum: G. F. Bolling, Westinghouse Electric Corp., T. B. Massalski, Mellon Institute, and C. J. McHargue, Oak Ridge National Lab.

The deformation stacking-fault probability, α, has been determined by the deformation of bulk specimens of zone-refined lead at 4.2° and 77°K. Aluminum (99.996 pct) α-brass (70:30), lead—0.10 atomic pct silver and lead—20.0 atomic pct indium were also examined at 4.2°K. It is shown that a difference exists between aluminium and lead, the latter being copper-like in its value of α. The influence of increased deformation in increasing the value of α is demonstrated. Addition of indium to lead suppresses a measureable value of α which correlates with observations made on twinning in this alloy.

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Short Range Order Strengthening in Cu-Al Single Crystals: T. J. Koppenaal and M. E. Fine, Northwestern University

Yield points were observed in Cu-Al single crystals containing 5, 10 & 14 atomic pct Al and have been attributed to short range order, which is known to exist in these alloys.

The yield points, $\Delta\tau$, which were produced by annealing 1.5 hours at 250°C between each test, are highly composition dependent. At 77°K $\Delta\tau$ was 50, 167 and 506 gm per sq mm for the 5, 10 and 14 atomic pct Al crystals, respectively; this is in good agreement with the theoretical composition dependency predicted for short range order strengthening. $\Delta\tau$ is also rather insensitive to the testing temperature when tested below 200°K. Above this temperature $\Delta\tau$ increases with increasing temperature ($\Delta\tau_{300°K} \sim 2\Delta\tau_{77°K}$ for 14 atomic pct Al crystals). A large strain increment (4 pct shear strain for the 14 atomic pct Al crystals at 77°K) is associated with the yield points, further evidence for short range order strengthening.

The effect of strain rate on $\Delta\tau$ was investigated by testing and annealing at 210°C. In the 14 atomic pct Al crystals $\Delta\tau$ increased with increasing strain rate by lowering the lower yield point. This is an inverse strain rate effect since the flow stress of a metal generally increases with increasing strain rate. It is felt that at faster strain rates and shorter diffusion times, the steady state degree of local order across an active slip plane will be smaller and the force required to pass succeeding dislocations will be correspondingly less.

Activation energies of motion, ϵ_M, were determined for the 14 atomic pct Al crystals by observing the time necessary to produce various $\Delta\tau$'s at temperatures from 50°C to 0°C. In the initial stages of strain aging, $\Delta\tau = 10$ gms per sq mm, ϵ_M is ~0.7 ev ϵ_M increases to ~1 ev for $\Delta\tau = 100$ gm per sq mm. These are in good agreement with values obtained by Wechsler and Kernohan for annealing quenched and neutron irradiated 15 atomic pct Al single crystals.

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Refractory Metal Activities in Europe

9:00 am to 12:00 m, West Ballroom

(Sponsored by IMD Refractory Metals Committee)

Chairman: J. J. Harwood, Ford Motor Co.

Survey of Refractory Metals Research in Europe: R. I. Jaffee, Battelle Memorial Institute

Highlights of Sheffield Refractory Metals Meeting: B. S. Lement, Manufacturing Laboratories, Inc.

Report of AGARD Activities in Refractory Metals: N. E. Promisel, Navy Department, and W. J. Harris, National Academy of Science

Plastic Deformation II

9:00 am to 12:00 m, East Ballroom

(Based on Submission of Research Abstracts)

Chairmen: A. R. C. Westwood, RIAS, and D. Wilsdorf, University of Pennsylvania

Strain Rate Studies of Al-1.7 at. pct. Cu at Low Temperatures: J. G. Byrne and M. E. Fine, Northwestern University

In Al-1.7 atomic pct Cu heat treated to various structures, variation of the flow stress with strain rate over the range 10^{-3} to 10^{-5} sec^{-1} was measured at various temperatures from 4.2°K to 373°K. An apparatus similar to that described by Blewitt was used for the 4.2°K measurements. The specimens were all at least initially single crystals. The structures studied were (a) supersaturated solid solution prepared by reversion, (b) solid solution plus GP I, (c) solid solution plus GP II, (d) solid solution plus θ', and (e) solid solution plus θ. In all cases the data fall roughly on straight lines when the cumulative increase in flow stress is plotted against the log of the strain rate. From the slopes of these lines effective activation volumes can be determined. If only a single activated process exists, the activation volume, v, may be interpreted in a manner similar to that proposed by Seeger and equal to bdl where b is the Burger's vector, d the activation distance, and l the spacing between obstacles. Also, from the increase in flow stress extrapolated to 0°K due to the absence of thermal activation, following Seeger, an activation energy may be computed. In (d) and (e) above more than one thermally active process appears to be indicated. For θ' in particular a second process is thermally active at 373°K. It appears reasonable to interpret the data for (a), (b) and (c) in terms of a single activated process. The v's at 4.2°K are 0.5, 0.3, and 0.5 x 10^{-20} cu cm, respectively, and the U_o's are 0.16, 0.1, and 0.1 ev, respectively. These values are interpreted in terms of mismatch dislocations presumed to exist around the precipitates for cases (b) and (c) in terms of clusters of solute atoms for case (a).

This research was sponsored by the United States Air Force through the Air Research and Development Command under Contract No. AF 49(638)-524. †Formerly Graduate Student, Northwestern University, is now with Dow Metal Products Co., Midland, Michigan.

Mechanism of Surface Effects in Crystal Plasticity: J. J. Gilman, General Electric Co.

The multiplication of dislocations in many crystals occurs by the multiple cross-glide mechanism. Therefore, the flow stress and strain-hardening rate can be influenced by things that affect dislocation cross-glide. It is proposed that surface films and surface-active liquids modify plastic flow in crystals by changing the rate of cross-glide at a surface. When a dislocation cross-glides it leaves a step behind it on the surface. The energy of this step resists the cross-glide motion and the energy of the step depends on its environment. Thus the rate of cross-glide depends on the environment, and so does the plastic flow. Unlike previous proposed mechanisms, this one accounts for the fact that environment does not affect the elastic limit of a crystal, but only alters the further course of the stress-strain curve.

Cross-slip and Cross-climb of Dislocations Induced by a Locked Dislocation: J. C. M. Li, U. S. Steel Corp.

The nature of cross-slip of screw dislocations induced by a locked parallel screw dislocation, that of edge dislocations induced by a locked edge dislocation through climb, and the cross-climb of edge dislocations induced by another edge dislocation through slip, will be discussed in the light of the interaction of their mutual stress fields. When a dilute slip band meets a locked dislocation, three things will happen, amongst others: (1) The slip band will be scattered by the locked dislocation and create a region of no slip on the other side of it; such a region will be called the *zone of re-*

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pulsion. (2) Dislocations within a certain region of the slip band will be attracted and captured by the locked dislocation; such a region will be called the zone of capture. (3) Dislocations gliding on some original slip plane within a certain region of the slip band can be induced to change to another slip plane of the same system through slip or through climb; such a region will be called the zone of cross-slip. The distance between the original slip plane and the final slip plane will be called a step of cross-slip. The situation of a band of climbing dislocations encountering a locked dislocation is similar and one can also define in such a case the zones of repulsion, capture, and cross-climb, and the distance between the original climbing plane and final climbing plane, a step of cross-climb. The dimensions of all these zones and steps depend on the external stress and the critical conditions for slip and for climb. Quantitative discussion of these dimensions will be given together with detailed description of the paths. Application of these results to experimental observations will also be discussed.

Temperature Dependence of the Yield Processes in Polycrystalline Iron: Raymond Ekvall and Norman Brown, University of Pennsylvania

The various stages of yielding in iron were analyzed by a new experimental method. The stress-strain tests employed a broad band of strain sensitivities from 10^{-4} to fracture in such a way that almost any sequence of strains and strain sensitivities may be investigated. Thus, a large strain may be immediately followed by a very small strain.

It was possible to observe non-elastic behavior at stresses as low as 8000 psi. This initial stress at which dislocation movement could be detected was not temperature dependent between 200° and 77°K where the ordinary yield point exhibits a very strong temperature dependence.

The next stress level, σ_2, in the yielding process was associated with permanent plastic deformation and was very nearly the same as the proportional limit reported in other investigations. σ_2 was temperature dependent, it increased by 20,000 psi between 200° and 77°K when the dislocations were freed from their atmosphere by a prior deformation at low temperature, and when the dislocations were pinned by an aging treatment, the temperature dependence of σ_2 was further increased by an additional 20,000 psi. The temperature dependence of the flow stress was the same as the temperature dependence of σ_2 of prior deformed iron, the temperature dependence of the uper yield point was about the same as σ_2 for aged iron.

It is concluded that if there is an appreciable Peierls-Nabarro force in iron, it is less than 8000 psi and it does not contribute to the temperature dependence. Also, the temperature dependence of iron is partly associated with Cottrell locking and partly associated with a stress sufficiently large to generate dislocations or to move them a long distance through the lattice. Possibly the latter stress is determined by random solute atoms the cutting of dislocations, or jogs.

This work is supported by ONR.

Dependence of the Mechanical Properties of Dislocation-free Germanium on the Electronic Properties: J. R. Patel and A. R. Chaudhuri, Raytheon Co.

As contrasted to metal crystals, it has been found for the semiconductor element, germanium, that it is possible to relate the deformation characteristics to certain electronic properties of the crystals.

The effect of heavy doping with an n-type donor element or a p-type acceptor element on the maximum stress reached during the tensile deformation of dislocation-free germanium has been reported previously. The impurity effects are due to the fact that the velocities of dislocations in n-type arsenic doped germanium are higher than in undoped germanium and in p-type gallium doped germanium lower than in undoped germanium.

The velocity behavior is in turn accounted for by a dislocation intersection mechanism involving the formation of trails of vacancies which serve to act as a drag to the motion of screw components of dislocations. Since vacancies behave as acceptors in germanium, the type and amount of impurity present influences the ease of formation of the vacancy trails, and hence determines the resistance to the motion of the dislocations.

The Fermi level is also a function of the temperature and the type of doping of the crystals. From measurements of the concentration dependence of either the maximum stress or the velocity, at various temperatures, it has been found that information can be obtained on the intrinsic carrier concentration as a function of temperature, on the displacement of the Fermi level from the center of the energy gap and on the position of the vacancy energy level. These determinations from the mechanical measurements are in good agreement with the direct electrical measurements of these parameters.

Some Recovery Characteristics of Zone-melted Iron: J. T. Michalak, U. S. Steel Corp., and H. W. Paxton, Carnegie Institute of Technology.

The isothermal kinetics of the recovery of the flow stress of polycrystalline iron have been studied as a function of the amount of tensile deformation, the temperature of the deformation, and the time and temperature of the recovery anneal.

The recovery of the initial flow stress is characterized by: (a) a logarithmic time dependence for the range of time over which the major recovery changes occur; (b) an increasing activation energy with increasing recovery over the temperature range of 300° to 500°C; (c) a slight increase in the rate of recovery, but no increase in the maximum amount of recovery following increased amount of prestrain; (d) an increase in both the rate of recovery and the recoverable component of strain hardening after prestrain at −78°C.

The recovery of a flow stress other than the initial flow stress appears to follow the same general kinetics of recovery as the initial flow stress, but is less advanced.

An isochronal study of the recovery of single crystals of this same material revealed more sluggish recovery as compared to the polycrystalline material, and the formation of substructure at relatively low annealing temperatures. The recovery of the initial flow stress of single crystals to a level equal to or less than the initial flow stress of the unstrained condition has been observed after annealing at 800° or 900° C.

The results are discussed in light of the current theories of work hardening and annealing.

Mobile Dislocations, Dislocation Velocities and the Macroscopic Plastic Properties of Dislocation-free Germanium: A. R. Chaudhuri, J. R. Patel, and L. G. Rubin, Raytheon Co.

The macroscopic plastic properties of dislocation-free germanium crystals are explained through a knowledge of the numbers and velocities of dislocations formed during plastic deformation, as has been proposed for lithium fluoride by Johnston and Gilman.

The method of Stein and Low has been used to measure average dislocation velocities under various stress conditions. The effects of stress, temperature and impurities have been determined. Dislocation velocities are strikingly different in silicon and germanium from those found in LiF and in Si-Fe, in that the velocities become essentially independent of stress at velocities of 10^{-1} to 10^{-5} cm per sec in the temperature range 400° to 700°C.

Average dislocation velocities were also determined from measured values of dislocation density after deforming to the maximum stress, assuming that all dislocations were mobile. Velocities thus measured were lower than those obtained by stress pulsing, thus indicating that all dislocations are not mobile, in a sample that is being deformed. In fact for certain orientations only 5 pct may be mobile.

The temperature dependence, and the strain rate dependence, of the maximum stress are due to both the numbers and velocities of dislocations. However, the temperature dependence of the strain rate, for a constant maximum stress, is due only to the temperature dependence of the velocities of dislocations. In the shear stress range 0.5 to 10 kg per sq mm, the activation energy for this process is 1.5 ev, and is independent of stress. The delay times in germanium can be determined from a knowledge of the dislocation velocities, and the results are compared with the direct measurements of the delay times obtained by Penning and de Wind.

Mechanical Properties of Tellurium Single Crystals: R. J. Stokes, T. L. Johnston, and C. H. Li, Minneapolis-Honeywell Regulator Co.

Tellurium has a hexagonal crystal structure in which the atoms are covalently bonded along spiral chains parallel to the [0001] direction. The bonds which cross link the chains are partially Van der Waals and partially metallic in nature. Slip occurs in the $\langle 11\bar{2}0 \rangle$ directions over $\{10\bar{1}0\}$ Class I prism planes which are also the planes of cleavage. We have determined the mechanical behavior in tension of chemically polished single crystals having the three following orientations: tension direction parallel to [11$\bar{2}$0], parallel to [10$\bar{1}$0], parallel to [0001] direction.

When specimens are favorably oriented for slip over $\{10\bar{1}0\}$ planes (I and II) they are extremely ductile, exceeding 20 pct elongation, providing great care is taken in their preparation and handling. The CRSS is very low, ~200 psi. For orientation, two

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{10$\bar{1}$0} planes are equally inclined at 60° to the tension axis and the active slip plane rotates towards the plane of maximum resolved shear stress. This favors continued single slip. For 2, two slip planes are at 30° to the tension axis and reorientation during plastic flow raises the stress on the dormant plane. This favors duplex slip. Rates of work hardening (2 higher than 1) and slip band appearance for the two orientations are consistent with this difference in slip behavior.

For orientation 3 there is no shear stress over {10$\bar{1}$0} planes and these specimens are extremely strong. They can support normal stresses of 20,000 psi with no detectable plastic deformation. The deformation and fracture characteristics for each orientation will be correlated with bonding anisotropy.

This work was partially supported by ONR.

Plastic Deformation of InSb: I—The Temperature and Strain-rate Dependence of Yield Stress: M. S. Abrahams, Radio Corp. of America

The initial deformation characteristics of single crystal InSb having a [145] tensile axis and containing about 1.5 x 10^{5} surface sources per cu cm have been studied. The specimens were deformed at temperatures between 300°C and 515°C and at shear strain-rates between 1.94 x 10^{-4} sec^{-1} and 1.94 x 10^{-3} sec^{-1}. At constant strain rate, the yield stress, τ_y, decreased linearly with increasing temperature and followed the equation, $\tau_y = M(T_m - T)$, where $M = -d\tau_y/dT$ and T_m (=525°C) is the melting temperature of InSb. Therefore, all the curves of τ_y vs T are seen to intersect at the melting temperature. The quantity M is related to the strain rate, $\dot{\gamma}$, by the equation, $M = a \tanh b\dot{\gamma}$, where $a = 5.11$ psi°C^{-1} and $b = 4.65 \times 10^{3}$ sec, and therefore is seen to attain a saturation value at high values of the strain-rate.

A series of samples, called β-type samples, were also tested in which a deviation was observed from the linear variation of τ_y vs T of the previous α-type samples. In this β-type behavior, the decrease of τ_y with T was less for 452 < T < 472°C, and then for 472 ≤ T ≤ 515°C, the variation of τ_y with T increased so as to again intersect at the melting point.

Electrical samples, associated with each tensile specimen, were given heat treatments similar to the tensile specimens and quenched from the elevated temperature. The percentage change in carrier concentration, electrical resistivity and Hall mobility was determined at both 78°K and 300°K. The α-type samples showed no change; the β-type samples exhibited large changes in all properties at both temperatures.

A model is presented to account for both the α- and β-type behavior in terms of impurity-locked surface sources expanding under the restraint of a large Peierls-type stress.

Yield and Fracture Stresses of Silicon Single Crystals: W. D. Sylwestrowicz, Bell Telephone Laboratories.

Single crystals of silicon were subjected to tensile tests in the range of temperature from 25°C to 1380°C. Nearly perfect crystals as well as those with many dislocations were used in the experiments. All samples were loaded in the <111> direction and had lateral faces with {110} and {112} indices.

It was established that above approximately 500°C single crystals of silicon could be plasticly deformed. The value of plastic strain increases with temperature and is of the order of 30 pct at 1300°C. Upper and lower yield points were observed in the range of temperature where plastic deformation takes place. Up to approximately 1100°C, tests ended in cleavage fracture; above this temperature in rupture. The cleavage stress increases exponentially with temperature. Ultimate tensile stress and upper and lower yield stresses decrease with temperature. It seems that the appearance of upper yield point is related to the oxygen content in material. Samples made from material, produced by floating zone method, containing small amount of oxygen do not show at all or very small upper yield point. A very distinct slip band system, corresponding to {111} slip, was observed on the surface of plasticly deformed samples. All observations described above refer to both types of crystals. There was no noticeable distinction between nearly perfect crystals and those with many initial dislocations. An explanation is offered for the conditions leading to cleavage fracture and to its temperature dependence.

Powder Metallurgy I

9:00 am to 12:00 m, Pennsylvania Room East

(Based on Submission of Research Abstracts)

Chairmen: A. Karion, Alan Wood Steel Co., and W. V. Knopp, SKC Research Associates

Production of Ultrafine Metal Powders by Ball Milling with Grinding Aids: Max Quantinetz, R. J. Schafer, and Charles Smeal, National Aeronautics and Space Administration

Normally, ductile metal powders cannot readily be ground to sub-micron particle sizes because of agglomeration or welding phenomena which occur in the grinding mill. This paper will describe how nickel and several other metal powders have been ground to sizes as fine as 0.1 μ through the use of selected grinding aids, especially complex metal salts.

This work was started as a result of a search for fine metal powders for use in dispersion strengthening studies. Since ultra-fine powders were not available commercially, it was decided to attempt to produce them by ball milling. Various grinding aids (metal salts with different ion size, valence, and polarizability) plus conventional surfactants were employed. Ethyl alcohol, water, cyclohexane, n-heptane, and methylene chloride were used as grinding fluids. Milling was conducted with Cr, Fe, Cu, Ag and Ni powders of different particle sizes.

Generally salts proved more effective as grinding aids than surfactants. The effectiveness of a salt as a grinding aid could be related to its valence characteristics and the polarizability of its ions. All of the metal powders noted above were successfully ground to sizes less than 1 μ. The type grinding fluid employed was found to significantly effect the grindability of the metal powders.

Effects of Relative Particle Sizes of Nickel and MgO Powders on the Strength and High-Temperature Stability of Dispersion Strengthened Nickel-MgO Alloys.: R. J. Schafer, Max Quantinetz, and J. W. Weeton, National Aeronautics and Space Administration

Of the many variables that can be introduced during the fabrication of dispersion strengthened alloys by powder metallurgy techniques, metal powder sizes and the percent volume of the oxide phase are believed to have pronounced effects upon the type of dispersion obtained. This investigation was undertaken to study the effects of these two variables upon the dispersed phase structure and the stress rupture properties of the nickel-MgO alloy powder metallurgy products.

Nickel powders of 1, .4, and .2 μ were combined with MgO powders of less than .1 μ. The oxide was added to the mixture in quantities of 4, 12, and 20 volume pct. Hot pressing and extruding of all specimens was done at 2100°F and stress rupture tests were made at 1800°F. Electron and optical micrographic studies were made. Variations in particle sizes and interparticle spacing of the dispersed phase were studied and attempts were made to correlate these parameters with stress rupture properties. It was noted that a pickup of NiO was obtained during ball milling. It is believed that this impurity influenced the agglomeration of some of the oxide MgO in the specimens.

Effects of Variations in Carbon Content, Heat Treatment and Mechanical Working on Stress-rupture Properties of a Liquid Phase Sintered High Temperature Alloy: P. A. Clarkin, J. W. Weeton, and P. F. Sikora, National Aeronautics and Space Administration

From a research standpoint, powder metallurgy methods may be considered to be a means of achieving structures not obtainable using normal ingot and mechanical working practice. Structures normal in castings may sometimes be avoided when alloys are made by powder metallurgical techniques. Macro- and microsegregations of alloying elements may also be avoided and more efficient use of these elements can be made.

This investigation deals with the effects of variations in carbon content and structure on the stress rupture properties of a cobalt base heat resistant alloy, produced by liquid phase sintering of prealloyed powders. The alloy selected for study had a matrix composition equivalent to that of carbon free S816. Carbon content was varied from 0.1 to 1.0 wt pct.

Results showed that stress rupture properties equivalent to or better than those of cast or wrought commercial S816 could be obtained with the powder product. In addition, the powder metallurgy techniques produced specimens with microstructures such that forging was readily accomplished even with carbon contents as high as 1 pct. A good correlation between stress-rupture properties and microstructure was obtained in many cases.

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Some Effects of Variation of Hot Pressing Temperatures Upon the Agglomeration of MgO in a Dispersion Strengthened Nickel-MgO Alloy. R. J. Schafer, Max Quantinetz, and J. W. Weeton, National Aeronautics and Space Administration

One of the requisites of dispersion strengthened alloys made by powder metallurgy techniques is that the particles which constitute the dispersed phase remain finely dispersed. This investigation, which is one of a series of studies, is concerned with the effects of hot pressing temperature on the agglomeration of MgO particles in a nickel-MgO powder compact.

The hot pressing temperature employed prior to extrusion in fabricating a dispersion strengthened nickel-MgO alloy was varied from 1800° to 2400°F. The nickel powder sizes employed were also varied from 0.1 to 1.0 μ. Oxide powder size was less than 0.1 μ in all instances. It was found that for a given volume percent of oxide, and a given extrusion temperature, large differences in agglomeration of oxides were observed with variations in either hot pressing temperature or nickel powder size.

Production of Refractory-coated Fine Metal Powders Suitable for Use in the Production of Dispersion-strengthening Alloys: W. B. Tarpley, C. D. McKinney, W. E. Marceau, Aeroprojects Inc., and G. S. Ansell, Rensselaer Polytechnic Institute

A method is described by which fine metal particulates are coated with thin layers of refractory compounds. These powders are sufficiently fine and the coatings adequately adherent to provide good starting materials for the production of dispersion-strengthened alloys by powder metallurgical techniques. The metal powders which have been coated include nickel and tantalum. These powders, in micron sizes, have been thinly coated with both aluminum oxide and chromic oxide. Electron micrographs of these coated particles are presented in which both the metal particles and their coatings are clearly evident. These powders were compacted, sintered, extruded into rods, and their metallurgical structures examined by electron microscopy. Quantitative evaluation thereof shows dispersion spacings of the second-phase particles in the alloy matrix ranging from approximately 2 to 5 μ. The dispersed refractory particles are flake-shape and comprise about 3 to 5 pct of the volume fraction of the alloy.

Further application of this technique for producing coated metal particles is discussed in relation to other two-phase systems which should be suitable as the starting material for dispersion-strengthened alloys.

The research here reported was sponsored by the Department of the Navy, Bureau of Naval Weapons.

New Metal-bonded Graphite Materials: M. Humenik, Jr., R. L. Van Alsten, and D. W. Hall, Ford Motor Co.

This paper comprises a discussion of the surface tension, liquid-solid interfacial energy, and wettability of Fe-C and Fe-C-Si alloys in contact with graphite. A process has been developed for controlling the wettability of graphite, which permits liquid phase sintering techniques to be employed for fabricating metal-graphite materials, ranging from 90 to 40 volume pct graphite. Data will be presented on the properties of these materials.

Powder Metallurgy Luncheon

Open to all who are interested in or wish to learn more about the subject

Time:	12:15 pm, Thursday, October 20
Place:	Tiered Balcony
Price:	$4.00
Tickets:	Purchase at Registration Desk

Engineering Fundamentals of the Rolling Process

9:00 am to 12:00 m, Pennsylvania Room-West

(Sponsored by IMD Shaping and Forming Committee)

Chairmen: W. A. Backofen, Massachusetts Institute of Technology

Present Status of Theory: L. F. Coffin, Jr., General Electric Co.

Boundary Conditions at the Roll-Material Interface: G. W. Pearsall, Massachusetts Institute of Technology

Resulting Structure and Properties: W. R. Hibbard, Jr., General Electric Company

Precipitation and Dispersed Phases

9:00 am to 12:00 m, Hall of Flags

(Based on Submission of Research Abstracts)

Chairmen: R. F. Bunshah, New York University, and W. B. Scott, Janney Cylinder Co.

Determination of the Number of Particles per Unit Volume From Measurements Made on Random Plane Sections: The General Cylinder and the Ellipsoid: R. T. DeHoff and F. N. Rhines, University of Florida

The number of intersections, N_A, that a random plane may be expected to make with N_V particles dispersed in an opaque matrix is

$$N_A = N_V \bar{D}_T$$

where $\bar{D}_T$ is the average distance between tangent planes for all orientations of all particles. It is possible to relate $\bar{D}_T$ to the average values of measurements which may be performed upon metallographic sections through the structure, for certain classes of aggregates of particles, and thus to compute N_V from measurements on random plane sections. These relationships had previously been derived for aggregates of spheres, rods, and plates of constant size; for spheres and platelets of arbitrary size distribution, and for cylinders of constant size and shape. The present paper extends this list of classes of aggregates for which N_V may be determined to aggregates of (1) circles, (2) particles with n flat circular ends, (3) cylinders of arbitrarily mixed shape and size distributions, and (4) ellipsoids of revolution of arbitrary size distribution.

It is found that, for the classes of shapes thus far studied, a formula of the type

$$N_V = k_I \alpha N_A \bar{Z}$$

results in which

k_I is a proportionality constant

α is a shape factor which may be determined from planar measurements

N_A is the number of intersections per unit area

$\bar{Z}$ is the average value of the reciprocal of some characteristic dimension of a planar cut.

Acknowledgement is accorded the United States Atomic Energy Commission, which has supported the program of research in Sintering of which this research has been a part.

Nucleation Catalysis by Carbon Additions to Magnesium Alloys: V. B. Kurfman, The Dow Metal Products Co.

Grain refinement of Mg-Al alloys by carbonaceous additives has been attributed by earlier workers to nucleation by Al_4C_3.

The work described demonstrates that many of the process and alloy variables can be interpreted and predicted from the known chemistry of the nucleating phase.

The grain coarsening effects of Be, Zr, Ti, RE, chlorination, temperature extremes and prolonged holding times are shown to

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depend on the chemistry and dispersion of the nucleant, and the requirement of a chemically clean nucleating surface in a dynamic system.

Conclusions are drawn stressing the chemical and dispersion criteria for successful grain refinement.

Nature of Nucleation of Precipitates in the Silicon-Lithium System: J. W. Ferman and R. A. Swalin, University of Minnesota

The kinetics of the precipitation of lithium in silicon have been studied in ultra high-purity silicon produced by the floating zone technique. Electrical resistivity techniques have been used to follow the progress of the precipitation reaction. Samples have been prepared containing lithium covering a range in concentration of two orders of magnitude. Lithium was introduced into the wafers at 700°C and the precipitation kinetics were followed at 95°C. A constant temperature of saturation has been used in order to minimize the number of variables influencing the process.

The precipitation process is characterized by rather pronounced transient effects after which the lithium precipitates according to a simple exponential decay law. From the exponential decay part of the curve values of τ, the characteristic precipitation time, are obtained and τ is evaluated analytically as a function of C_o, the initial lithium content. It is found that

$$\tau^{-1} \sim [C_o]^{1/3}$$

From the concentration dependence of τ several interesting points concerning the nucleation phenomenon can be deduced. (a) Lithium is not precipitating homogeneously since the influence of lithium concentration on τ is too small. (b) A critically sized nucleus for precipitation is not an Li_4O^+ cluster as has been suggested. This follows since according to the law of mass action

$$4\,Li^+ + O = Li_4O^+$$

and hence the number of nuclei (Li_4O^+) should be proportional to C_o^4. This concentration dependence is not observed but rather it appears that the number density of nuclei is independent of C_o. This conclusion, arrived at on the basis of kinetics, has been confirmed microscopically by use of an etchant developed in our laboratory for selectively delineating the lithium-rich precipitate particles. With this etch, it has been determined that precipitate particles are platelike in shape and lie on {111} type planes of silicon.

In order to determine whether or not oxygen is playing an active role in the nucleation process, experiments were performed in which aluminum was introduced into the samples prior to the introduction of lithium. Since it is known that aluminum forms ion pairs with dissolved oxygen in silicon, this treatment would reduce the particle number density of lithium precipitates if oxygen is important in the basic nucleation reaction. A reduction was indeed found to be the case.

The nature of the precipitation catalyst and the role of oxygen in the process will be discussed.

This research was sponsored by the Office of Naval Research.

Microstructure of Unidirectionally Solidified Al-$CuAl_2$ Eutectic: R. W. Kraft and D. L. Albright, United Aircraft Corp.

Solidification experiments were conducted with the objective of testing the theory of eutectic colony formation. The results of tests on a series of high-purity Al-Cu specimens of eutectic composition indicate that the formation of eutectic colonies can be suppressed by appropriate control of the impurity content, the solidification rate, and the thermal gradient in the liquid at the liquid-solid interface. A unique microstructure consisting of lamellae essentially parallel to one another throughout a large test specimen was produced. The three-dimensional morphology of growth imperfections observed in these specimens was determined by examining successively polished surfaces. The defects, termed lamellar faults, were found to originate from extra lamellae. Lamellar faults, which are morphologically similar in many respects to edge dislocation models in crystals, produced a complex substructure in these specimens.

In addition to the faulted lamellar microstructure and the colony microstructure (in which, incidentally, faults were also observed), a third type of eutectic, a banded structure, was observed under certain growth conditions. The reasons for the appearance of this microstructure are not completely understood but certain influential growth parameters have been tentatively established.

Structural Relationships Between Precipitate and Matrix in Cobalt-rich Cobalt-Titanium Alloys: R. W. Fountain, G. M. Faulring, and W. D. Forgeng, Union Carbide Metals Co.

The mechanism of precipitation of the fcc phase, Co_3Ti, from supersaturated solid solution of a γ-cobalt-6 pct titanium alloy has been investigated using single-crystal X-ray diffraction techniques. Solution-heat-treated single-crystal slices were aged for various times at temperatures of 600° and 800°C. The aged crystal slices were oriented with their [100] or [110] directions parallel to the incident X-ray beam.

Oscillation patterns of specimens aged for short time periods at 600°C show satellite reflections indicative of a coherent precipitate parallel to the {100} matrix planes. After 48 hr at 600°C, the discrete satellites merge with the matrix reflections. This is attributed to partial loss of coherency of the precipitating phase with the matrix. Aging for 48 hr at 800°C results in the formation of two distinct fcc phases identified as Co_3Ti (a_o = 3.603A) and a solid solution of cobalt and titanium (a_o = 3.573A. The Co_3Ti phase precipitates in the same crystallographic orientation as the matrix.

In the initial stages of aging at 600°C., transmission Laue patterns show unusual reflections in the small-angle regions. Based on double-Bragg reflections and variable parameters of the matrix, a hypothesis is presented to explain this small-angle phenomenon and to corroborate the observation on the coherent growth of the Co_3Ti phase indicated by the satellites on the oscillation patterns. The progress of growth of the precipitate with aging time indicated by changes in the characteristics of the Laue reflections of the matrix is correlated with changes in the hardness of single crystals as well as the electrical resistivity of polycrystalline specimens of similar composition.

Electron microscope studies of the precipitate morphology confirm that the precipitate is in the form of thin plates, as deduced from the X-ray diffraction studies. Electron diffraction analysis of the precipitate extracted from aged alloys substantiates that the Co_3Ti phase is ordered.

Electrolytic Dispersion Hardening: W. H. Meiklejohn and R. E. Skoda, General Electric Co.

A process of dispersion hardening will be described which consists of the electrodeposition of an insoluble metal into a liquid base metal so as to form a fine particle dispersion of the insoluble metal. The particular system of copper containing 1 μ particles of molybdenum will be discussed.

The electrodeposition was carried out at 1200°C in an argon atmosphere. An anode of molybdenum was suspended in a fused electrolyte of barium and magnesium fluorides. The cathode was a molten pool of copper. High frequency heating was used so as to cause some stirring of the metal. The melt was chill cast to obtain a uniform distribution of the molybdenum particles.

The increase in the yield strength due to the dispersion of molybdenum particles and the temperature dependence of the yield strength are in agreement with the predictions of the equations developed in our previous work on the Fe-Hg system.

Copper wires containing 1.4 volume pct of Mo particles were annealed at 500°C and measured to have a yield strength of 21,000 psi at room temperature. The 100 hr creep to rupture strength at 450°C was 2-½ times that of pure copper. The resistivity was 8 pct greater than gfc copper at room temperature but the difference gradually decreased with increasing temperature until at 200°C the resistivities became equal and remained equal up to 400°C.

Effect of Temperature on the Yielding Behavior of Several Aluminum-Aluminum Oxide Dispersion-strengthening Alloys: F. V. Lenel, G. S. Ansell, and R. A. Bosch, Rensselaer Polytechnic Institute

The yielding behavior of several alloys, consisting of a matrix of commercial purity aluminum containing a finely dispersed second-phase of aluminum oxide was determined as a function of temperature, over the temperature range from − 90°C to 500°C. The proportional limit of these materials was used as the criterion for yielding. The observed variation of the proportional limit with temperature is in good agreement with the behavior predicted by the theory proposed by Ansell and Lenel to describe the yielding behavior of dispersion-strengthened alloys, where the yield strength at a temperature T, σ_T, varies with temperature in the following manner:

$$\sigma_T = \sigma_m \left(\mu_T \mu^*_T / \mu_m \mu^*_m\right)^{1/2}$$

where σ is the yield stress, μ and μ^* are the shear moduli of the matrix and the dispersed phase respectively, and the subscripts T and 25 refer to values of these at test temperature and at 25°C.

The support of this research by the National Aeronautics and Space Administration is gratefully acknowledged.

Precipitation of Carbon From α-Iron: R. H. Doremus, General Electric Co.

The carbides initially precipitating from α-iron containing less than 0.3 wt pct C were observed with the electron microscope,

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and the rate of this precipitation was followed from the internal friction of the iron. For aging temperatures between 60° and 170°C both studies indicated that the carbides precipitated on dislocations in the iron in both strained and unstrained material. In unstrained samples with about .022 wt pct C the rate of precipitation below 60°C was anomalously high, indicating that many more nuclei than could fit on the dislocations were present. From the kinetic study and for other reasons it was concluded that the equation of Cottrell and Bilby for the segregation of atoms to dislocations was not valid for the precipitation of carbon from strained iron, and that previous agreement between this equation and experiments was fortuitous. From an electron diffraction pattern the carbide phase was tentatively identified as having the bct structure of an expanded α-iron lattice with about 0.7 wt pct C. This phase seemed to be unstable, transforming into cementite or ϵ carbide when the iron was over-aged. No satisfactory theory for the rate of precipitation has been proposed, although this process can be understood qualitatively. Furthermore no explanation is available for the lower activation energy of growth as compared to that for the diffusion of carbon in α-iron which was found in a previous study.

Solubility and Precipitation of Nitrogen in Iron Containing Manganese: J. F. Enrietto, Jones & Laughlin Steel Corp.

The fact that manganese inhibits the precipitation of nitrogen in α Fe has been established, but quantitative solubility data for nitrogen in Fe-Mn alloys are lacking. This investigation was designed to quantitatively determine the effect of small manganese additions on the solubility and precipitation rates of nitrogen in α iron.

Internal friction techniques, following quench aging, were used to determine the solubility of nitrogen in equilibrium with some nitride. Equilibrium was approached from both the supersaturated and undersaturated states. Since both means of approach yielded the same result it was apparent that true equilibrium had been attained. Five sets of alloys were tested, the manganese concentrations being about 0.03, 0.1, 0.3, 0.7, and 0.9 wt pct. The alloy containing 0.3 pct Mn may be considered typical of the effect of manganese on the solubility of nitrogen. The heat of solution over the temperature range 200°-550°C was about 5,800 cal per mole for this alloy. This can be compared with a value of about 8,000 cal per mole for nitrogen in pure α iron. The precipitation process was extremely sluggish; for example, over 200 hr were required for complete precipitation at 200°C to take place. In binary Fe-Ni alloys, at the same temperature, the process requires only a few minutes. It might also be noted that no reversion phenomena were observed in the Fe-0.030 pct Mn alloy. It took, in fact, several hours to dissolve the precipitates at 346°C. This indicates that the precipitates are very coarse and that manganese somehow inhibits the nucleation of nitride particles.

Effect of Prior Heat Treatment on Carbon Strain Aging in Low Carbon Steels: J. F. Butler, Jones & Laughlin Steel Corp.

In annealed low carbon steels containing no nitrogen in solution, strain aging is affected by variations in the cooling rate from austenite prior to sub-critical annealing. The effect was studied in plain Fe-C alloys and in boron low carbon steels where the nitrogen was present as BN. Low frequency internal friction was used to measure the amount of carbon in solution; the amount of strain aging was measured by the increase in the homogeneous flow stress at 7.5 pct prestrain after an aging treatment of 1 hr at 212°F. Carbon strain aging in the Fe-C alloys and boron low carbon steels can increase from 0 psi at a cooling rate of 500°F per min to 6,000 psi at a cooling rate of 1°F per min. The increased degree of strain aging at slower cooling rates was due to an increased amount of carbon in solution.

The dependence of carbon strain aging and the amount of carbon in solution upon the cooling rate from austenite is explained by the dispersion of carbides formed. Water quenching or air cooling through the A_1 results in a carbon supersaturated ferrite which produces a fine carbide dispersion during sub-critical annealing. Slower cooling rates through the A_1 result in the formation of widely spaced pearlite colonies which are changed little by subsequent annealing. On slow cooling from the annealing temperature, the carbon diffusion distance to the finely dispersed carbides is small so that carbon can be effectively removed from solution whereas the large distances between pearlite colonies prevents effective carbon removal and allows strain aging to occur.

Refractory Metals

2:00 to 5:00 pm, West Ballroom

(Based on Submission of Research Abstracts)

Chairmen: H. W. Schadler, General Electric Co., and R. W. Fountain, Union Carbide Metals Co.

Mechanism of Purification of Tungsten Rods by Electron Beam Floating Zone Melting: H. G. Sell, Westinghouse Electric Corp.

The growth of long single crystals of tungsten of very high purity by the electron beam floating zone method is strongly affected during the first and second pass by the impurity concentration of the starting material. Heavy contamination makes a successful growth practically impossible and single crystals which are grown from impure material at a rate of 2-4 mm per min. still contain micropores.

The mechanism of pore formation and pore elimination was studied by forming stationary partial molten zones and completely molten zones which were kept molten for different times. The molten zones were then cut along the rod axis and perpendicular to it and the surfaces were investigated by photomicrography.

It was found that micropores form during the first melting. These pores migrate along the liquid-solid interface towards the axis of the rod. They coalesce into small bubbles during migration and finally segregate into a larger bubble at the axis of the molten zone. The big bubble continues to grow when the zone is moved. If the impurity concentration is too high the big bubble grows to a critical size and sudden burn-through occurs. In very pure starting materials one medium size bubble each is segregated at the starting and finishing end.

Photomicrographs demonstrating the bubble formation will be presented and the mechanism is interpreted in terms of insoluble impurities. Bubble volume and impurity concentration are compared. The bubble stability is explained energetically.

Tensile Properties of Arc-melted Tungsten and Tungsten-base Alloys at Temperatures Up to 1925°C: E. L. Harmon, Union Carbide Metals Co.

The tensile properties of impact-extruded and swaged arc-melted tungsten were determined over a range of temperature from 25° to 1925°C. A peak in the yield strength and ultimate tensile strength occurred at about 500°C., accompanied by a minimum in reduction in area. This behavior, as well as the occurrence of a maximum in the elongation versus temperature curve at slightly lower temperatures, may have been caused by strain aging. At temperatures of 1650° and 1925°C., arc-melted tungsten exhibited high values of elongation and reduction in area, in contrast to swaged powder metallurgy tungsten, which exhibited a substantial decrease in ductility at these temperatures. Testing at a faster strain rate improved the ductility of the powder metallurgy tungsten at 1650°C.

Tensile properties of tungsten binary alloys containing small additions of titanium, zirconium, and columbium were determined at 1650° and 1925°C. The ultimate tensile strength of each of the alloys was noticeably higher than that of unalloyed tungsten, with a 0.57 pct Cb alloy displaying the greatest strength (60,600 psi uts at 1650°C.). The strengths of the alloys were related to the degree of recrystallization and the grain size. There was no apparent correlation of strength with oxygen or carbon content. No second phase was observed in the alloys.

This work was partially supported by W.A.D.D.

Substructure, Recrystallization Behavior, and Ductility of Tungsten: E. L. Harmon and W. D. Forgeng, Union Carbide Metals Co.

The effect of annealing times and temperatures upon the microstructure of swaged tungsten rod was studied. Concurrent changes in the ductile-to-brittle transition temperature of the material were also noted. The variations in transition temperature, which could not be explained completely by the grain structures observed by usual metallographic procedures, appeared to result from changes in substructure. These substructures were only revealed by vacuum thermal etching. Very fine, diffuse subboundaries produced better ductility than coarse, sharply defined subboundaries. From these observations, it appears that the well-known embrittlement that accompanies the recrystallization of cold-worked tungsten can be rendered less severe by introduction of a suitable substructural configuration.

This work was partially supported by W.A.D.D.

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Recovery in Cold-worked Tungsten as Affected by Impurities: R. C. Koo, Westinghouse Electric Corp.

Recovery in high purity, cold-worked tungsten rods was investigated by means of a dislocation etch-pitting technique, supplemented by X-ray back reflection, hardness and tensile measurements. In spite of the high melting point of tungsten, it was found that the dislocations were highly mobile at relatively low annealing temperatures. Upon annealing at 800°C the high density of dislocations initially present fairly at random in the as-worked material was reduced to a density of ~ 10^6 per sq cm and a highly polygonized substructure was clearly evident. Increase in annealing temperature caused progressive subgrain coarsening accompanied by extensive grain coarsening of the originally deformed grains above 1100°C (strain-induced boundary migration). The addition of approximately 30 wt ppm of Fe had no observable effect but the addition of approximately 30 wt ppm of Al had a strong effect in retarding polygonization and subgrain coarsening and raised the recrystallization temperature by about 350°C. However, it was found that strain-induced boundary migration was insensitive to impurities at these levels.

The effect of aluminum on the various mechanisms of recovery and the dependence of mechanical properties on subgrain size will be discussed.

Yield Point in Tungsten Single Crystals: R. C. Koo, Westinghouse Electric Corp.

In an experiment on the deformation of electron-beam zone-refined tungsten single crystals at subzero temperatures, it was observed that the as-grown crystals did not exhibit a sharp yield point even though the crystals contained sufficient quantities of interstitial impurities for forming a Cottrell atmosphere for all the dislocations present. Annealing the virgin crystals or the prestrained crystals in vacuum at various temperatures did not alter the behavior. A sharp yield point, however, was always observed on crystals annealed in a wet hydrogen atmosphere independent of the amount of prestrain. If a wet hydrogen annealed crystal was first strained and then aged in vacuum, the yield point again appeared upon reloading. Furthermore, the introduction of the yield point was always accompanied by an increase in flow stress and the magnitude was found to increase with increase in the water vapor content in the annealing hydrogen. Other tests showed that the water vapor was only partially responsible for the effect observed.

Vacuum fusion analyses of interstitial impurities failed to yield any correlation with the mechanical tests. The results of many other tensile tests on crystals annealed under different conditions will be described and the origin of the yield point observed will be discussed.

Influence of Zone Refining on the Brittle-to-ductile Transition Temperature of Polycrystalline Tungsten: J. L. Orehotsky, General Telephone and Electronics Laboratories, Inc.

Tungsten rods were zone refined, cold worked, and recrystallized to various grain sizes. The brittle-to-ductile transition temperature was determined and compared with values obtained for the original unzoned recrystallized rods. The zone-purified, worked, and recrystallized material had a significantly lower transition temperature than the original unzoned samples of similar grain sizes. The transition temperature was also found to be dependent upon the grain size of the material.

Effect of Structure and Purity on the Mechanical Properties of Columbium: A. L. Mincher and W. F. Sheely, Union Carbide Metals Co.

The effects of structure and impurity content on the mechanical properties of columbium have been studied over the temperature range of −196° to 1093°C. It was found that the strengthening influences of cold work and impurities may decrease with temperature below ambient. An interpretation of this phenomenon has been offered. Above ambient temperature, the strengthening effects of cold work persist almost unchanged up to 400°C above which they progressively decrease. Maxima in the rate of strain hardening during tensile tests in certain specific elevated temperature ranges arise from strain aging by interstitial impurities. Analysis of the data has revealed the temperature ranges at which carbon, oxygen, and nitrogen, respectively, are responsible for the strain aging. Also, on the basis of this analysis, it has been possible to suggest which interstitial atoms are responsible for the evidences of strain aging that have been reported for vanadium and tantalum.

Effect of Grain Size on the Oxidation of Columbium: W. T. Hicks, E. I. duPont de Nemours and Co.

Little difference in rate of reaction between coarse and fine grain columbium and dry oxygen at 1 atm was observed from 450° to 1200°C using continuous gravimetric techniques. However, examination of specimens reacted to 50 pct of completion revealed some unexpected details of the oxidation mechanism: (1) Extensive grain boundary penetration occurred in the coarse grain specimens from 450° to 650°C; such penetration also occurred in the fine grain material but to a lesser extent. (2) A fine, intragranular penetration, possibly along planes of preferred orientation, was observed in both coarse and fine grain material from 500° to 670°C. (3) Samples reacted at 700°C and higher temperatures had comparatively smooth surfaces and no grain boundary penetration.

The relatively uneven attack of columbium surfaces may account for the break-away type oxidation curves and maximum in the rate previously reported for columbium in this temperature range.

Creep, Fatigue, and Fracture

2:00 to 5:00 pm, East Ballroom

(Based on Submission of Research Abstracts)

Chairmen: H. Conrad, Atomics International and M. Schwartz, Frankford Arsenal

Steady-state Creep Behavior of a Dispersion-strengthened Aluminum Oxide Alloy With Coarse Dispersion: G. A. Ansell, F. V. Lenel, and J. C. Yen, Rensselaer Polytechnic Institute

The investigation of the steady-state creep behavior of SAP type alloys previously reported was extended to an alloy having a larger mean free path between oxide particles. This alloy, designated RP 15-30 has an average spacing between oxide particles of 6μ. The steady state creep rate was determined as a function of stress and temperature.

In the stress range where dislocations can bow between the dispersed particles leaving residual loops the stress and temperature dependence of the creep rate is the same as previously found for the alloy AT 400 with a finer dispersion, i.e. the creep rate is proportional to the fourth power of the stress and the logarithm of the creep rate is inversely proportional to the absolute temperature with an activation energy equal to that of self diffusion of aluminum. The ratio of the creep rate of the RP 15-30 alloy to that of the AT-400 alloy is equal to the square of the ratios of the dispersion spacing. These relationships are in accord with the creep theory proposed by Ansell and Weertman.

At lower stresses where dislocations cannot bow between the dispersed particles, Ansell and Weertman's theory postulates a creep rate proportional to stress. Such a relationship was established for the creep of the RP 15-30 alloy at low stresses, although for alloys with finer dispersion the creep rate at low stress drops rapidly to immeasurably small creep rates in this stress region.

The support of this research by the National Aeronautics and Space Administration is gratefully acknowledged.

Grain Boundary Sliding in Lead Bi-crystals: P. R. Strutt, Franklin Institute Laboratories and R. C. Gifkins, University of Melbourne

The bi-crystals were grown in vacuo from hydrogen-reduced high-purity lead; the boundary was straight and at 45° to the tension axis. Sliding was measured using the offsets at marker lines and creep was measured using sets of gauge marks near to and distant from the boundary. Temperatures from 21°-110°C and stresses of 100-400 psi were used. Metallographic observations were made throughout the tests. There was no qualitative difference between sliding measured parallel to the stress axis or transverse to it. Mean sliding correlated linearly with creep extension across the boundary; in this zone slip systems operated in addition to the primary ones in the component crystals. Several methods of obtaining activation energies were used, giving scattered values; however, all methods gave values for sliding similar to that for creep in the boundary zone, but less than for creep of the whole specimen. Boundary migration was usually absent or slight, except in localized areas. Alternative interpretations of the observed stress dependence are discussed.

Direct Measurements of the Surface Energies of Crystals: J. J. Gilman, General Electric Co.

By means of quantitative cleavage, the surface energies of several simple crystals have been measured at −196°C. The crystals

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and their cleavage planes are: LiF (100), MgO (100), CaF_2 (111), $CaCO_3$ (1010), Si (111), Zn (0001); their respective surface energies (ergs per sq./cm) are: 340, 1200, 430, 230, 1240, 105. The measured values for LiF and MgO are in good agreement with simple ionic theory.

The cleavage surface energy (an effective quantity that is affected by plastic deformation) of LiF is only moderately dependent upon increasing temperature. The behavior of MgO is similar. In contrast, the cleavage surface energy of Zn increases rapidly with increasing temperature as non-basal plastic glide increases. Fe (3 pct. Si) crystals also exhibit rapidly increasing cleavage surface energy with increasing temperature.

A small amount of cadmium (0.1 atomic pct.) markedly increases the cleavage surface energy of zinc.

Mechanical Behavior of AL_2O_3 Whiskers at 25°C to 2030°C: S. S. Brenner, General Electric Co.

The tensile strengths of sapphire whiskers grown by the method first described by Webb and Forgeng have been measured as a function of temperature, time and environment. The average short time (~1 sec) strengths, $\bar{\sigma}$, of the whiskers decrease roughly linearly from about 900,000 psi at 25°C to 150,000 psi at 1965°C. The standard deviation from the mean was found to be as high as $0.4\bar{\sigma}$.

In static loading, the strengths of the whiskers is time dependent decreasing approximately 10 pct for each order of time. The delayed fracture occurs both in hydrogen and oxygen and it is therefore unlikely that it is caused by an environmental interaction. Plastic deformation prior to fracture was not detected although the occurrence of small amounts cannot be ruled out. Except at the very highest temperature, the fracture was of the brittle type.

The temperature and time dependence of the strengths of the whiskers has as yet not been rationalized on theoretical grounds. The Griffith mechanism of crack growth and failure provides a temperature dependence that is several times smaller than the observed one.

Fracture Behavior of Magnesium Oxide Bi-crystals: A. R. C. Westwood, Research Institute for Advanced Studies.

Metallographic studies on compressed bi-crystals of magnesium oxide have revealed that grain boundaries are extremely effective barriers to the movement of edge dislocations, causing them to pile-up at the boundary. Often cracks are associated with such piled-up groups, but these are produced only when two slip bands, one from each grain, meet at the boundary. No cracks associated with a single piled-up group of dislocations have been observed. Crack propagation is transgranular and by crystallographic cleavage. Other observations indicate that grain boundaries can serve as a prolific source of dislocations.

A mechanism for crack formation at low strains is proposed in which a suitably oriented source, situated on the opposite side of the grain boundary and some two to eleven microns away from a pile-up group of edge dislocations, is activated by the stress field associated with this pile-up. The source then emits dislocations in the direction of the boundary but of opposite sign to those in the activating group. These dislocations are also arrested by the boundary causing a second pile-up which is usually displaced a small distance (~1 μ) from the primary group. Under certain conditions, the two piled-up groups may then co-operate to produce a crack in the grain boundary.

Metallographic evidence for the mechanism will be presented.

Effect of Cyclic Loading on Magnesium Oxide Crystals: A. J. McEvily, Jr., National Aeronautics and Space Administration, and E. S. Machlin, Columbia University

MgO crystals have been tested in reversed bending in order to determine their behavior under cyclic loading. Single crystal specimens were cleaved and chemically polished prior to testing. It was found that under constant amplitude reversed loading fatigue failures did not occur in these crystals even at stresses twice as high as the initial yield stress. (However, for a sufficiently large amplitude of plastic strain, cracks could be nucleated at dislocation pileups during the first reversal of strain). The absence of fatigue failures in further evidence of the importance of cross slip in fatigue, since cross slip is a difficult process in MgO crystals.

Incidental to the main purpose of the investigation it was found that chemically polished MgO crystals exhibited no aging effect with time. It is considered that the fatigue failures and aging effects of MgO crystals that have been reported by other investigators are associated with microcracks in the surface of cleaved but not polished crystals.

Formation of Fatigue Cracks in Aluminum Single Crystals: T. H. Alden, General Electric Co., and W. A. Backofen, Massachusetts Institute of Technology

Pure aluminum single crystals of a fixed orientation were cycled at constant deflection in reverse pure bending and observations made of strain hardening and slip-band formation.

Initial hardening for the usual strain amplitude of 0.2 pct was complete within about 50 cycles, subsequent plastic strain giving first a zero and then a very low hardening rate. Fine lines from primary slip formed in increasing numbers during rapid hardening. Annealing the fine slip continued through large numbers of cycles. Only after hardening to saturation did slip cluster in the way normally associated with fatigue straining. At saturation fine, widely spaced lines form on a polished surface. These intensify gradually with cycles both by spreading and by forming a notch-peak cross section, at the same time becoming fragmented and nonuniform. Material elevated above the original surface level, termed extrusion, develops steadily at a rate of about 1-2 μ per thousand cycles. Fatigue cracks form in the resulting slip bands.

A thick and unbroken anodic film, applied by electropolishing, prevents completely the formation of fatigue cracks. Any break in this film becomes the source of failure, while metal underlying the intact film remains uncracked. Removing cracks in the film with a brief polish permits a substantial increase in life.

Using an anodic film to prevent surface notches, it is shown that fatigue cycling does not internally weaken the slip bands. Slip-band cracking is concluded to be the result of active slip in the presence of free surface. The hardening behavior and observations of slip-band development are consistent with the idea that cross slip is important in establishing conditions suitable for fatigue cracking.

† This paper is based on a thesis by T. H. Alden submitted to the Massachusetts Institute of Technology in partial fulfillment of the requirements for the degree of Doctor of Science.

* General Electric Research Laboratory, Schenectady, New York.

** Associate Professor, Department of Metallurgy, Metals Processing Laboratory Massachusetts Institute of Technology.

Suppression of Cleavage Fracture of a Cobalt-Iron Alloy. C. W. Chen, Westinghouse Electric Corp.

Two methods have been revealed for the suppression of cleavage fracture of a Co-Fe alloy. The first method requires the formation at grain boundaries of a discontinuous network of precipitates resulting from the fcc→bcc phase transformation. Low cohesion at the interface between the lattice and precipitates gives rise to interfacial cracking in place of {100} cleavage fracture. The second method relies on the generation of immobile dislocations upon the collapse of discs of quenched-in vacancies in fcc structure. The immobile dislocations impede the motion of slip dislocations, thereby preventing large piling up of dislocations at the grain boundaries. Thus, by appropriate annealing and quenching, the fracture of the alloy shifts from the inherent cleavage type through a mixed shear, cleavage, and intergranular mode to a ductile mode.

Supplementary electrical resistivity measurements and specially designed precipitation experiments support the theory that dislocations are generated from quenched-in vacancies in specimens exhibiting high ductility. The results of this investigation also imply that {100} cleavage cracks are initiated in the present case by the piling-up of dislocations against grain boundaries (Stroh's mechanism), rather than by the interaction of slip dislocations on two {110} planes (Cottrell's mechanism).

Study of Fracturing Behavior of Metals Coated with Mercury: N. A. Tiner, Douglas Aircraft Co.

A study was made of the effects of type of loading, temperature and electric polarization on the fracturing behavior of mercury coated zinc and copper. It was noted that zinc showed mercury stress cracking in both static and cyclic loading, whereas copper showed the effect only in cyclic loading. The effect was present in polycrystals as well as in single crystals.

The sub-zero testing revealed a transition temperature for the mercury embrittlement. The effect was completely eliminated in zinc at the temperatures below 70°F. The embrittlement of both zinc and copper was slightly reduced if the mercury coat was made cathodic relative to the base metal.

Microscopic examination revealed a continued penetration of mercury into the grain boundaries and propagation of cracks along these continuous paths in zinc as observed by others. Moreover, the electron microscopic examination of replicas from the fracture surface showed that in mercury coated copper fatigue cracks propagated at high velocities by a mechanism of local transcrystalline shear.

It was concluded by Smith, Robertson and others that the basic fracturing process in mercury coated metals is controlled by the lowering of the solid-liquid interface tension at the grain boundaries. The observations reported in this paper suggest that although

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the grain boundary penetration by the wetting effect may play a role in the initiation of cracks in static type of loading. It is not an essential requirement for crack propagation. The mercury embrittlement is due principally to the lowering of the energy absorption with crack growth by the interaction of chemical and mechanical effects. Cracks may propagate along the continuous path of grain boundaries, as well as the basal slip planes or the tip of fatigue crack embryos.

Powder Metallurgy II

(Based on Submission of Research Abstracts)

2:00 to 5:00 pm, Pennsylvania Room East

Chairmen: J. D. Shaw, SKC Research Associates, and H. Hirsch, General Electric Co.

Sintering of Very Fine Oxide Particles Produced by Calcination of Salts: K. Iwase, T. Takada, and T. Hayashi, Kyoto University, Japan.

Electron microscopic and diffraction studies have been made of the mode of sintering of Fe_2O_3, Cr_2O_3, CoO and reduced Fe powders prepared by calcination of the respective metallic salts. When $(NH_4)_2Cr_2(SO_4)_4 24H_2O$ is heated below 750°C, the formation of Cr_2O_3 proceeds very slowly and a well developed large crystal is often obtained. When the salt is calcined at higher temperatures, large crystals are seldom obtained but rather an aggregate of very fine Cr_2O_3 particles. In general, the size of the individual particles increases as the calcining temperature increases. This is explained by the sintering of the particles during calcination. Electron diffraction studies indicate that the crystallographic axes of the individual oxide particles in an aggregate produced from a single crystal of salt are frequently aligned. This may be due to the similarity in the distribution of metal ions in the mother salt and in the oxide.

An Examination of the Early Stages of Sintering of Metal Particles: Marvin Herman, G. E. Spangler, and Henri Amar, Franklin Institute Laboratories.

The early stages of the sintering of metal particles have been examined by measuring the rate of densification of uncompacted spherical metal powders heated with the temperature increasing at a uniform rate. With the slow heating rates used expansion due to gases escaping from the pore volume has been virtually eliminated and thus the values of the dimensional changes obtained relate only to particle neck growth. The sintering equations which describe the kinetics of shrinkage based on mass transport by diffusion have been modified to account for the non-isothermal treatment; the following expression has been derived considering that temperature is linearly related to time:

$$F(S) = \frac{CD_s}{a} \exp\left(-\frac{MQ}{RT_i} - b\right)$$

where,

$F(S)$ = some parameter relating to neck growth

$C = \dfrac{}{B\gamma^{1/3}/k}$ B = constant depending upon diffusion model

γ = surface energy

δ = interatomic distance

k = Boltzmann's constant

D_s = frequency factor in diffusion

Q = activation energy for diffusion

R = gas constant

T_i = instantaneous temperature

a = heating rate

m and q = constants relating to the solution of an exponential integral.

The analysis of the results of the shrinkage during sintering of iron and copper spherical powders indicate that the process proceeds by a diffusion mechanism. In addition metallographic evidence will be presented which in the case of iron clearly shows the dependence of shrinkage on the presence of grain boundaries.

This work has been sponsored by the Atomic Energy Commission.

Nickel Activated Sintering of Tungsten Powder Compacts and Sprayed Deposits: J. H. Brophy, L. A. Shepard, and J. Wulff, Massachusetts Institute of Technology.

Experimental evidence has been presented recently by the authors showing that nickel-coated hydrogen-reduced tungsten powder may be sintered to 98 pct of theoretical density at 1100°C. Simultaneously a theory was presented by which this process may be explained. According to this postulate, the nickel coating serves as a carrier phase in which tungsten atoms are free to move. The kinetics of the process closely resemble those observed when the carrier phase is a liquid, although in the present case, there is no evidence of the existence of a liquid phase either in equilibrium phase relationships or in microstructure at the temperatures under consideration.

This analysis has been extended to the case of nickel tungsten powder co-reduced from the oxide and subsequently sintered. Data in the literature by other investigators reveal the existence of nickel activated tungsten sintering in raw material prepared in this way, but the explanation presented for the mechanism is qualitative and not well established. The present work attempts to show that the first stage of sintering of the co-reduced powder is kinetically similar to that observed for coated powder, with outstanding differences arising from the more closely defined location of the nickel carrier in the latter case. The role of grain growth in the second stage of sintering in both cases is important.

The technique of nickel activated tungsten sintering has been successfully extended to the densification of plasma sprayed coatings. Densities of the order of 95 pct of theoretical are obtainable at 1300°C. Theoretical analysis again suggests kinetics like those of nickel-coated powder sintering with modifications due to a considerably different particle shape. The practical implications of the process are discussed.

The research reported here was supported in part by the Bureau of Naval Weapons, Department of the Navy, Materials Division.

Kinetics of Growth of Aluminum Oxide Particles in a Nickel Matrix: F. V. Lenel, J. Dromsky, and G. S. Ansell, Rensselaer Polytechnic Institute

Alloys consisting of a matrix of nickel containing a finely dispersed phase of Al_2O_3 were prepared by coprecipitating the mixed hydroxides of nickel and aluminum from aqueous solution, calcining the precipitate to a mixture of oxides, selectively reducing the NiO to nickel, compacting the powder and resistance hot pressing the alloy.

Electron micrographic studies were made of the alloy after annealing at temperatures in the range from 1150°C to 1300°C for times up to 100 hr. The dispersed phase of Al_2O_3 was observed to coarsen as a function of time at annealing temperature.

Quantitative electron microscopy was used to determine the kinetics of this coarsening process.

The support of this research by the National Aeronautics and Space Administration is gratefully acknowledged.

Shrinkage Behavior of Copper Compacts and Loose Powder Aggregates: F. V. Lenel, H. H. Hausner, E. Hayashi, and G. S. Ansell, Rensselaer Polytechnic Institute.

The relative shrinkage in the radial and in the axial direction of conventionally pressed copper powder compacts and of loose copper powder aggregates was investigated. Irregular electrolytic copper powder, atomized spherical copper powder and flake copper powder were studied. The structure of the green and of the sintered compacts and aggregates was examined metallographically. The results of the experimental work lead to the tentative conclusion that the theories advanced to explain the differences in the axial and radial shrinkage of compacts and aggregates are not correct. The pores of green and sintered compacts of irregular copper powder are not disk or lens shaped, but equiaxed and the difference in shrinkage can therefore not be attributed to the shape of the pores. On the other hand, the ratio of axial and radial shrinkage of irregular powder and of flake aggregates is nearly the same in spite of the fact that the pores in the flake powder aggregates are much larger in the radial than in the axial direction. The possibility that stresses in the plastic region have a much greater influence upon shrinkage behavior than pore shape is discussed.

The support of this research by the Research Division of the United States Atomic Energy Commission is gratefully acknowledged.

Textures, Recrystallization, and Recovery

2:00 to 5:00 pm, Pennsylvania Room West

(Based on Submission of Research Abstracts)

Chairmen: J. J. Becker, General Electric Co., and E. R. Levin, Frankford Arsenal.

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Secondary Recrystallization in High-Purity α—Iron: C. G. Dunn and J. L. Walter, General Electric Co.

Secondary recrystallization is found to occur in high-purity α-iron in a manner that is quite similar to secondary recrystallization in high-purity 3 pct silicon iron. That is to say, after primary recrystallization and some normal grain growth certain grains with the lowest surface energy at the gas-metal interface grow to become secondaries. In thin iron sheet annealed in dry hydrogen the low energy plane is (110); the secondary recrystallization texture is (110) [001].

Experimentally, cold-rolled strip 0.0025 in. (0.065 mm) thick was produced from an ingot of high-purity electrolytic iron. The final reduction in thickness was 75 pct. The final sheet specimens were annealed at 885°C in an atmosphere of hydrogen in which the H_2O/H_2 pressure ratio was maintained near 10^{-4}.

The present results, contrary to recently published work, indicate that impurities are not required for stabilization of the matrix structure. Instead the necessary stability may arise from grain boundary thermal grooving associated with the *thickness effect*.

Pierre Coulomb, Metaux, 35, 1 and 66 (1960).

Specific information on the role of orientation dependent surface energy is obtained through a study of grain boundary interactions. An effect of orientation on thermal faceting is noted. Information is also obtained on the formation of annealing twins during both primary recrystallization and secondary recrystallization.

Secondary Recrystallization Kinetics in Singly Oriented Silicon Iron: T. V. Philip and R. E. Lenhart, Crucible Steel Co. of America.

Singly oriented Si-Fe obtains its (110) [001] texture by a secondary recrystallization process. The nature of the initial and steady state growth kinetics of these secondary grains from the fine grained primary matrix is the main subject of this paper. In addition, the location of the secondary nuclei and the nature of their initial growth inhibition by a precipitate dispersion are discussed.

It has been observed that successful secondary recrystallization requires that primary grain growth be prevented. Primary grain growth apparently destroys the small number of grains that can serve as growth nuclei for the secondary grains. The effectiveness of the primary grain growth inhibition is strongly reflected on the final grain size and magnetic properties of the material.

In observing the nature of this secondary recrystallization process, the activation energies for the start of secondary grain growth and for the continued growth of the secondaries have been measured. The activation energies are 103 Kcal per mole and 75 Kcal per mole respectively. In addition, a model is proposed to explain the high activation energy observed for the initial growth of the secondary grains. The model involves the solution and redistribution of the primary grain growth inhibiting precipitate.

Photomicrographs are shown that reveal the location of the (110) [001] nuclei in the cross section of the material. The photomicrographs also reveal the course of the secondary grain growth and show it to be driven by grain boundary energy.

Growth of (110) [001] Oriented Grains in High-Purity Silicon Iron—A Unique Form of Secondary Recrystallization: J. L. Walter and C. G. Dunn, General Electric Co.

Recent reports have discussed the formation of the cubic texture secondary recrystallization and the (110) [001] texture by tertiary recrystallization in high-purity silicon iron. The driving force for grain growth in both cases has been shown to derive from differences in the energy of the various crystallographic planes at the gas-metal interface. The energies of these various {hkl} surfaces are not constant but vary with the amount of impurity atoms adsorbed at the surface. Thus, in a slightly oxidizing atmosphere, γ_{100}, the energy of the (100) surface, is lower than γ_{hkl} (hkl is any surface other than 100). In a non-oxidizing atmosphere (for material initially low in oxygen) $\gamma_{100} < \gamma_{hkl}$.

The present report discusses the formation of the (110) [001] texture by secondary recrystallization which occurs when high-purity 3 pct Si-Fe, initially low in oxygen, is annealed in a non-oxidizing atmosphere.

Samples were prepared by rolling vacuum melted ingots to sheet 0.012 in. thick. The material contained less than 0.0006 Wt pct Ox and about 0.005 pct total impurities. Electropolished samples were annealed in vacuum at 1200°C. The (110) [001] oriented matrix grains grew to become secondaries. Any departure from the conditions of non-oxidizing atmosphere and low oxygen content resulted in growth of (100) oriented grains.

Nuclei for secondary recrystallization, contrary to the usual relationship, correlate directly with the strongest component of the primary matrix texture, (110) [001]. From observations of grain boundary migration during secondary recrystallization, the relationship, $\gamma_{110} < \gamma_{hkl}$ was obtained. Oriented growth is not involved in the secondary recrystallization.

Growth Rates of Surface Energy Controlled Secondary Grains in 3 pct Silicon Iron Sheets: K. Foster, J. J. Kramer, and G. W. Wiener, Westinghouse Electric Corp.

An investigation of the effect of primary grain size and sheet thickness on surface energy controlled secondary growth rates of (100) grains in 3 pct silicon iron sheets was made, where secondary grain growth was carried out under conditions in which (100) plane had the lowest surface energy. To describe the results, the relation

$$G = M\left(\frac{\gamma_B}{r} + \frac{2\,\Delta\gamma_S}{t} + C\right)$$

was derived; here G is the secondary growth rate, M is the mobility of the grain boundary, γ_B is the grain boundary energy, r is the average primary grain radius, $\Delta\gamma_S$ is the difference in surface energy between the (100) plane and the average primary matrix, t is the sheet thickness, and C is a negative driving force related to the holding up of grain boundaries by inclusions. Growth rate measurements of (100) secondaries were made for four primary grain sizes and two sheet thicknesses (essentially all other variables were held constant). A value for the ratio $\Delta\gamma_S/\gamma_B$ of about 3 pct was obtained, indicating that the grain boundary energy supplies most of the driving force for growth. The energy supplied by the surface energy difference is required for secondary growth, but primarily only to select the secondary grain and to trigger the secondary growth process.

Recrystallization of Iron and Iron-Manganese Alloys: W. C. Leslie, F. J. Plecity, and J. T. Michalak, U. S. Steel Corp.

Isothermal recrystallization and grain growth in zone- and vacuum-melted irons and Fe-Mn alloys, up to 0.60 pct Mn, were studied in the range 480°-650°C, after 60 pct cold reduction.

In the early stages of recrystallization, the rate of grain growth and rate of recrystallization of high-purity iron decreased with time at constant temperature. The recrystallization of heavily cold-rolled, polycrystalline, high purity iron is consistent with the proposal that recrystallization is a growth-controlled process, with nucleation occurring at substantially zero time.

It is proposed that, in the early stages, the rate of grain growth is highly strained, polycrystalline, high-purity iron is proportional to the residual strain energy of the deformed matrix. This, in turn, is determined by the extent to which recovery has occurred. The characteristic kinetics of recovery determine the kinetics of grain growth. In the early observable stages of isothermal crystallization, the linear rate of grain growth, G, may be represented by the equation

$$G = K_1 F_o - K_2 \ln t$$

where F_o is the residual strain energy at zero time, t is time, K_1 and K_2 are constants. After recovery ceases, the growth rate becomes constant, until affected by impingement.

Up to a concentration of about 0.30 wt pct Mn reduces the rate of growth if primary recrystallized grains according to the relationship

$$G = G_o \exp(-K \cdot \text{pct Mn})$$

where G_o is the rate of growth in high-purity iron. Above about 0.30 pct Mn, the rate of growth of recrystallized grains is only slightly reduced by additional increments of manganese. The mechanism of recrystallization changes at about 0.30 pct Mn. At this point, nucleation becomes a function of time. Although rate of grain growth decreases with time, the rate of recrystallization increases.

In comparison with other elements, manganese has a comparatively small effect upon the recrystallization of iron.

Recovery of Cold Worked Aluminum and Copper under Stress: P. H. Thornton, University of Pennsylvania, and R. W. Cahn, University of Birmingham, England.

For most cubic metals, there is no convenient metallographic method for distinguishing recrystallized from unrecrystallized ma-

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terial during the annealing process. A microhardness technique has been described which enables the degree of recrystallization to be determined for 99.999 + pct cu. In this method a measure of the amount of recrystallization which has occurred is made from the splitting of the microhardness histogram into two peaks, one corresponding to the unrecrystallized material and the other to the recrystallized material. This provides a means of determining the effect of stress on the recovery and recrystallization behaviour of copper. Experiment showed that only a very slight recovery effect could be produced by a stress under conditions which did not lead to rapid creep failure or recrystallization; the main effect of stress was to cause work hardening of the recrystallized grains. Although copper of lower purity, when partially recrystallized, did not produce these duplex peaks in the microhardness histograms, a similar conclusion was reached.

Experiments were carried out on 99.99 pct Al to determine by how much, softening could be produced, by recovery alone, when annealed under stress. Hardness was again chosen as a measure of the amount of recovery; the annealing temperature was selected such that no recrystallization could occur with the annealing times employed. The presence of stress during the annealing process increased both the kinetics and the extent of recovery. The amount of recovery could be increased from about 18 pct to over 50 pct and this increase could be correlated with the creep deformation produced.

This work was sponsored by A.E.R.E., Harwell, England, as part of an investigation on recrystallization under stress.

Effect of Orientation on the Recrystallization Kinetics of Cold Rolled Single Crystals: W. R. Hibbard, Jr., and W. R. Tully, General Electric Co.

Single crystals of copper and silicon-iron were cold rolled in orientations chosen to produce individually the major components of the polycrystalline deformation texture. The orientation dependence of the kinetics of recrystallization was studied in relation to the textures produced by primary recrystallization of these specimens. It is not possible to rationalize the polycrystalline recrystallized textures on this basis. The results suggest an important role of oriented growth and the interaction of adjacent crystals during the deformation of aggregates.

Effect of Annealing Temperature on Preferred Orientation in High-purity Lead: K. T. Aust and J. W. Rutter, General Electric Co.

During a study of the mobility of grain boundaries in bicrystals having controlled orientations, it was observed that the temperature dependence of grain boundary migration in zone-refined lead is smaller for large angle coincidence-type grain boundaries than for large angle non-coincidence *random* boundaries. For example, the mobility of coincidence boundaries is greater than that for random boundaries at 175°C, but their relative mobilities are similar at 300°C.

The above observation suggested the following experiment, which was conducted to further evaluate the role of grain boundary mobility in the development of preferred orientation. New grains, introduced by localized deformation at room temperature followed by annealing at 300°C or at 175°C, were allowed to grow competitively at the expense of the substructure of melt-grown single crystals of zone-refined lead. The orientation relationship was determined between the melt-grown crystal and the recrystallized grain which grew most successfully in each specimen. After annealing at 300°C, the only distinguishing feature is the fact that the grain boundaries which are able to migrate successfully out of the deformed region are always large-angle boundaries, i.e., random orientation relationships are obtained. However, after annealing at 175°C, the most successful grain boundaries separate crystals which have a coincidence-site orientation relationship, i.e., preferred orientations are obtained.

These results demonstrate, in an unequivocal manner, the importance of the temperature dependence of grain boundary mobility on the development of a preferred orientation in an annealed, high-purity metal. The observed coincidence boundaries were of the type considered by Kronberg and Wilson, Dunn, and Frank, and separate crystals which are related by certain rotations about <111>, <100> and <110> axes.

Annealing Twins in Zone-refined Aluminum: K. T. Aust, General Electric Co.

The present study deals with the nature of the interfaces information of annealing twins during the growth of recrystallized grains into striated, melt-grown single crystals of zone-refined aluminum used in this work was characterized by an electrical resistivity ratio between room temperature and 4.2°K of about 9000.

It was found that where an annealing twin was formed during grain boundary migration, a large angle random boundary was replaced by either a large angle coincidence boundary, a near-twin boundary or a medium angle boundary. The coincidence boundaries observed in the high-purity aluminum separated crystals having orientation relationships corresponding to a relatively high density of coincidence sites. In a previous study on zone-refined lead by Aust and Rutter, the formation of annealing twins resulted in the appearance of coincidence boundaries corresponding to low as well as high densities of coincidence sites. These observations are consistent with the larger ratio of twin boundary energy to grain boundary energy in aluminum as compared to lead.

The present results support the Fullman-Fisher theory of annealing twins and provide evidence in favor of the interpretation of certain large angle grain boundaries in terms of the coincidence site model.

Temperature Dependence of Rolling Textures in High Purity Silver: Hsun Hu and R. S. Cline, U. S. Steel Corp.

The rolling texture of silver is commonly known as {110} [$\bar{1}$12], whereas that for most of the other fcc metals, such as Cu, Al, Ni, etc., is mainly {123} [412]. A texture transition from the *copper-type* to the *silver-type* can be effected by solute additions. Among many fcc alloys, α brass is a typical example for such texture changes.

A basic understanding of the formation of the two different types of rolling textures in a fcc lattice has long been lacking. Smallman has suggested that slip-plane hardenability and *overshooting*, which are dependent on both concentration and temperature, may be responsible for the texture transition in alloys. However, just how *overshooting affects* the final orientation at high deformations is not at all clear. Brown, incorporating the ideas of Seeger and of Hirsch, has suggested the dependence of wire textures on stacking fault energy. To check this idea, we conducted an investigation to see whether a texture transition can be produced in high-purity silver by varying the rolling temperatures, and subsequently, to correlate any change in texture with stacking fault frequency.

The silver used is of such high purity (total metallic impurities spectrographically detected <2 ppm; O_2, 2 ppm) that substantial recrystallization takes place at room temperature after 40-50 pct reduction. Therefore, a low-temperature device must be used for texture determinations. The texture determined at 0°C for a specimen rolled 91 pct at 0°C was the normal {110} [112] type. Hence, the relatively high oxygen content of common silver is probably not responsible for the formation of the {110} [112] texture as Smallman suggested. The texture determined at room temperature for a specimen rolled at 200°C was primarily of the *copper-type*, together with a cube-texture component. Microscopic examination indicated partial recrystallization, presumably due to reheating the strip between the last few rolling passes. The cube-texture component obviously arises largely from the recrystallized grains.

The present results are in accord with the observations of Müller, who obtained a {110} [112] texture in copper rolled at a low temperature. Thus, the change of rolling texture from one type to the other in fcc metals can be obtained by merely varying the temperature of deformation. Further experiments are currently in progress at this laboratory.

Evolution of Textures in FCC Metals, Part III: Correlation With Silver Chloride: Y. C. Liu and R. H. Richman, Ford Motor Co.

The plastic deformation modes of AgCl have been reported as similar to those in fcc metals. In order to provide additional insight into the origin of preferred orientations in fcc metals, the deformation and recrystallization textures of AgCl have been studied in detail. Reagent grade AgCl carefully processed to avoid contamination develops a rolling texture almost identical to that of copper. Well-defined pole concentrations are centered at the {135} <211> orientation, which is different from the ideal orientations suggested in the past for the deformation texture of copper. The spread of deformation orientations also extends to the {110} <112> position. When the AgCl is contaminated with SiO_2 during processing, the peak height ratio technique reveals a transition of the deformation texture toward the 70:30 brass type.

Annealing uncontaminated cold-rolled AgCl at low temperatures produces a recrystallization texture consisting of the {110} <001> ± 8° orientation, similar to Cu-Sb solid solution alloys of high antimony content. No {001} [100] component is detected immediately after the completion of primary recrystallization. Some effects of varying the annealing temperature and time are also presented and discussed.

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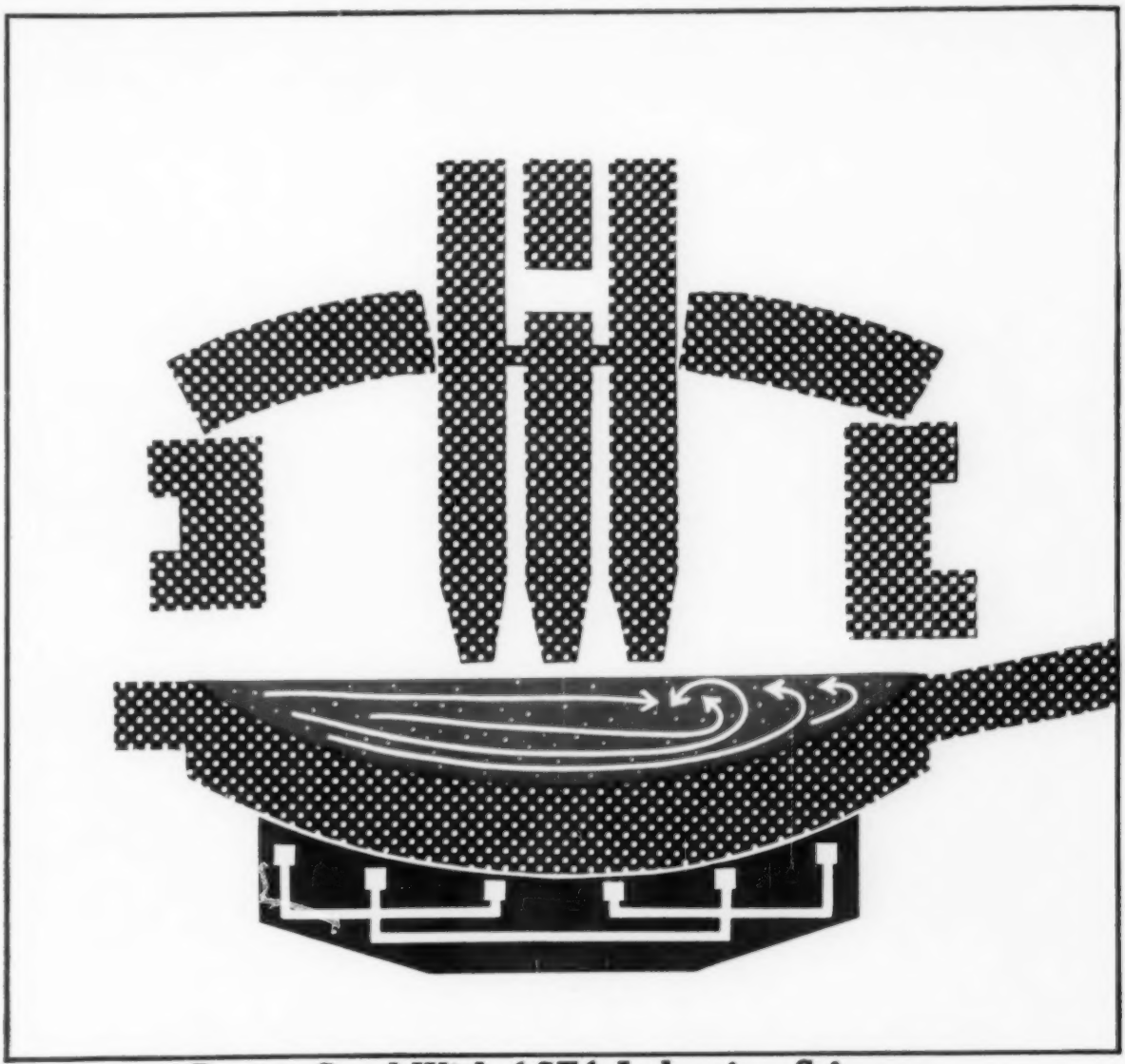

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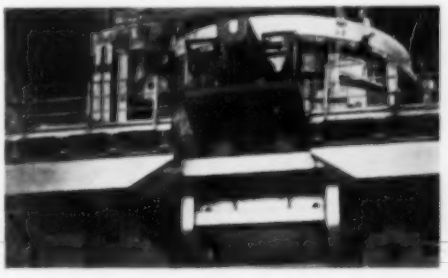

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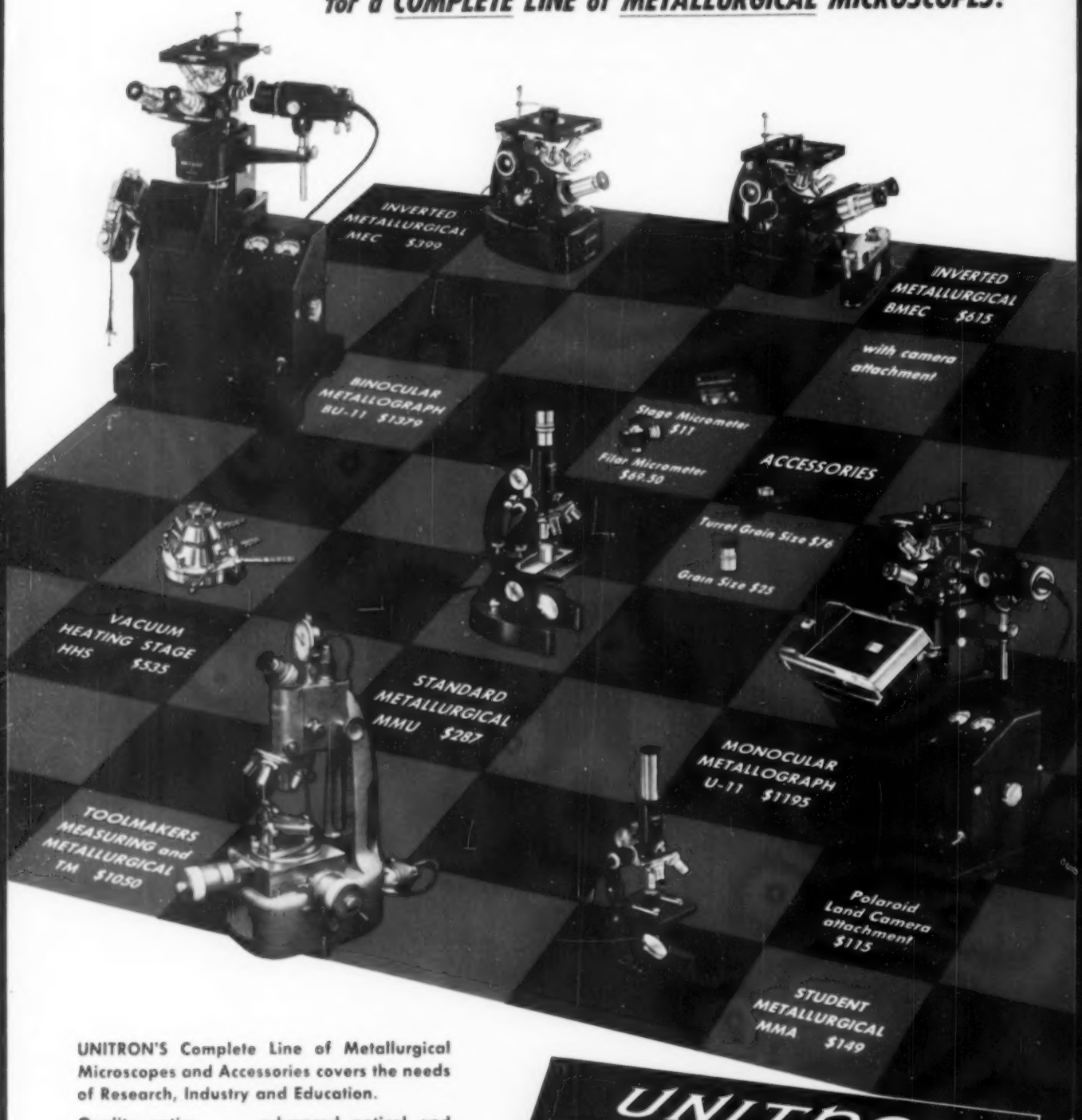

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